4.2.6 Exposure of Amphibians to Asbestos

4.2.6.1 Data That Are Valuable for Evaluating Effects of LA on Amphibians

Amphibians may be exposed to LA in the aquatic environment (including exposure to both water and sediment), and also to LA in soil in terrestrial environment. Of these two environments, it is suspected that the highest exposure and the greatest susceptibility is likely to occur during the early (aquatic) life stages of this receptor group, so attention is focused on aquatic media (i.e., surface water and sediment). The following lines of evidence are all potentially useful in evaluating risks to amphibians from LA in surface water and/or sediment:

- The computational HQ approach: measurement of LA concentrations in site waters and sediments, interpreted by comparison to appropriate TRV values
- *In-situ* measurements of effects: measurement of malformation frequency in metamorphs in the field
- Site-specific population studies: measurement of amphibian population density and diversity in the field
- Site-specific toxicity tests: Measurement of toxicity to selected life stages in laboratory-based toxicity tests using site water and/or sediments
- LA toxicity tests: Measurement of toxicity to selected life stages in laboratory-based spiking studies using LA added to laboratory water and/or sediment

4.2.6.2 Summary of Existing Data

At present, there are no data from OU3 to support any of the lines of evidence potentially useful for evaluating the risks to amphibians from LA in surface water or sediment. Measures of LA concentration in water and sediment from OU3 are available, but there is no suitable TRV for LA toxicity in either medium for amphibians.

4.2.6.3 Data Quality Objectives for Amphibians

Step 1: State the Problem

Historic mining and milling operations at OU3 have resulted in the release of LA to the environment, including surface water and sediment in ponds within OU3. Amphibians may be

exposed to LA in these environmental media during their aquatic life stage via direct contact and ingestion. The problem being investigated is: Do exposures to concentrations of LA in site sediment and water result in significant reductions in survival, growth or metamorphosis in site specific amphibian toxicity tests? Reproduction was considered as a separate endpoint, but the length of time required, 5-6 additional months, and resources needed to complete a full reproduction study were determined to be impractical to implement.

Step 2: Identify the Goal of the Study

The goal of the Phase III amphibian investigation is to determine if exposure of amphibians to LA in surface water and sediment in ponds in OU3 will result in ecologically significant adverse effects on survival, growth, or metamorphosis.

Step 3: Identify the Information Inputs

The information inputs that are needed to address the study goal include reliable measures of survival, growth, and metamorphosis in developing amphibians exposed to LA in water and sediment. Exposure levels should include LA values that are at the high end of the range of concentrations observed in OU3 ponds. Analogous data from amphibians exposed to uncontaminated water and sediment are also needed to allow for comparisons between contaminated and uncontaminated locations.

Step 4: Define the Bounds of the Study

Spatial Bounds

Amphibians breed primarily in ponds rather than flowing streams. Based on this, the areas of OU3 that are most likely to provide suitable habitat for amphibians include the Tailings Impoundment, the Mill Pond, Fleetwood Creek Pond and Carney Creek Pond. Testing will be conducted in the laboratory with site sediment collected from an OU3 pond containing concentrations of LA at the high end of the range detected in all ponds (e.g., the Tailings Impoundment) (spatial bounds).

Concentration Bounds

The concentrations of LA in surface water and sediment to be tested should be near or above the high end of the concentrations that have been observed in water and sediment in on-site ponds. Although there is variability in the environmental cues that influence the timing of breeding and metamorphosis for amphibian species that are likely to occupy OU3, the time interval of chief interest is from early May to September, the time period in which most amphibians are released from their gelatinous egg cases, become free swimming larva and undergo development and metamorphosis in the aquatic environment.

Data on surface water concentrations of LA in OU3 ponds during the period early May to mid July are summarized in Table 4-7. As seen, concentrations of LA in pond water over the time frame of interest range from non-detect (<0.05 million fibers per liter (MFL) to a maximum of 83 MFL (Fleetwood Creek Pond). However, based on studies performed to date in support of the OU3 RI, including a juvenile trout toxicity test performed using site waters in 2009 and analysis of surface water samples collected at stream sampling station LRC-06 in July 2009, it is now known that these measures of LA concentration may influenced by fiber clumping and/or binding to sample bottle surfaces. Thus, actual surface water concentrations may be higher. To be conservative, the water concentration to be tested will be 10 billion fibers per liter (BFL).

The concentrations of LA in sediment in the ponds are summarized in Table 4-8. As seen, the maximum LA concentration measured in all OU3 ponds analyzed by PLM-VE was 2%. Based on this maximum, site sediments will be collected from locations that will likely yield sediments with LA concentrations greater than or equal to 2%,

Step 5: Develop the Analytic Approach

The analytic approach is to measure ecologically relevant endpoints in amphibians exposed to LA in water and sediment at concentrations that represent the high end of on-site conditions, and to determine if these endpoints are statistically different from those measured in organisms exposed to control sediment and water. The following table summarizes the measurement endpoints and their relation to the assessment endpoints:

Assessment Endpoint	Measurement Endpoints
Survival	- Hatching success - % mortality - Incidence of malformations that could affect survival
Growth	- Whole body weight - Snout-vent length (SVL); whole body length - Incidence of malformations that could affect growth
Metamorphosis	-Time to developmental stage - Median Metamorphosis Time (MMT) - Hind limb length (normalized to SVL), if metamorphosis does not occur - Necropsy (internal and external) - Incidence of malformations that could affect development or reproduction -Tissue pathology (head, tail, serum) (if determined to be necessary)

Note that "incidence and severity of histological lesions in gonad tissue" was considered as a reproduction measurement endpoint but both the assessment and measurement endpoints for reproduction were replaced (as indicated in the table above) after considering that it would

require the toxicity test to continue for approximately 150 days to allow sufficient time for gonad development. This time period was considered to be impractical to implement.

The precise statistical tests that will be used to compare exposed and control organisms will vary between the measurement endpoints. For discrete endpoints (survival, malformation frequency), it is expected that comparisons will be made using the Fisher Exact test. For continuous endpoints (body weight, MMT), it is expected that the comparisons between control and treated groups will be performed using the Wilcoxon Rank Sum (WRS) Test (unless the data are distributed approximately normally, in which case comparisons may be performed using t-statistics). Other statistical tests that may be appropriate include one-way ANOVA or an ANOVA on ranks. *Post hoc* tests may also be used such as Dunnett's test or Bonferroni t-test for parametric sets, or Dunn's test for non-parametric tests. If these comparisons show significant differences or are inconclusive, additional histological investigations may be performed. To accommodate potential additional investigations, the body (head and tail) and serum will be preserved.

If no statistically significant differences in any of the endpoints are detected between the exposed and the control organisms, it will be concluded that exposure to LA in surface water or sediment at concentrations equal to or less than the levels tested are not likely to cause effects that are ecologically significant. If statistically significant changes in one or more measurement endpoints are observed, additional investigation may be needed to determine if those effects result in ecologically significant effects at the population level, to determine if the effect is caused by the water or the sediment, and to identify a no-effect level that may be used to evaluate remedial alternatives.

Step 6: Specify Performance or Acceptance Criteria

In evaluating the results of amphibian toxicity testing, two types of decision errors are possible:

- A false negative decision error occurs when it is decided that there are no significant effects on amphibians, when in fact there are
- A false positive decision error occurs when it is decided that there are significant effects on amphibians, when in fact there are not

As discussed in EPA (2002), the probability of decision errors when comparing two data sets (site *vs.* reference) is controlled by the selection of the null hypothesis, and by selection of an appropriate statistical method to test the null hypothesis. Two alternative forms of null hypothesis are possible:

- Form 1: The null hypothesis is that no difference exists between site and reference. A confidence level of $100(1-\alpha)$ % is required before the null hypothesis is rejected and it can be declared that the site data are higher than the reference data.

- Form 2: The null hypothesis is that the site is higher than reference by some amount (S) that is considered to be biologically significant. A confidence level of 100(1- α) % is required before the null hypothesis is rejected and it is declared that that the difference between site and reference, if any, is smaller than S.

For the purpose of this effort, the Form 1 null hypothesis is selected for use because it is the most familiar, is the easiest to interpret, and does not require specification of an effect that is presumed to be significant. In accord with EPA (2002), when the Form 1 null hypothesis is used, it is appropriate to select a value of α that is somewhat higher than the usual value of 0.05, such that marginal differences between site and reference are more easily identified as being significant. In accord with this, α is set to 0.20.

Step 7: Develop the Plan for Obtaining Data

A detailed protocol for the amphibian toxicity study will be developed by the toxicity testing laboratory and submitted to EPA for review and approval. Table 4-9 summarizes important features of the amphibian toxicity test that will be performed. Key features are discussed below.

Study Design

The target exposure concentrations of LA in surface water (10 BFL) and in sediment (2%) could be achieved either by collecting on-site media of the appropriate concentration levels, or by adding ("spiking") LA to control media. Based on a consideration of the potential complexities of collecting sufficient quantities of on-site surface water media with the appropriate concentration levels, as well as the potential for problems caused by microbial growth and LA adherence in sample collection bottles, the spiking approach with static renewal is judged to be the most appropriate for surface water used in this investigation. Because field biological and environmental fate processes are difficult to duplicate, the collection of on-site sediment from known, high end locations is judged to be the most appropriate approach for test sediments.

Based on this strategy, the study design will include three groups:

Group	Sediment	Water
1	Synthetic sediment	Laboratory water
2	Reference (uncontaminated) field sediment	Laboratory water
3	Contaminated field sediment (approx. 2% LA)	Laboratory water spiked with LA

Each exposure group will consist of four replicate exposure chambers each containing 20 organisms. Embryos will be assigned to exposure chambers at random. The study protocol will specify how embryos will be assigned to control/treatment groups.

Exposure chambers will be 9.5L aquaria fitted with standpipes to provide a tank volume of 6 L. Aquaria temperature will be maintained at 20-23°C. A static-renewal design will be used. The frequency of water changes is not yet known, and will be determined based on pilot studies. It is anticipated that a renewal frequency of once every 2 to 3 days may be needed to maintain DO levels >3.5 mg/L as well as account for LA adherence to tank walls.

The test sediments will be added to each tank and will cover the bottom to a depth of 2 cm. The expected volume of sediment required for each exposure tank is approximately one liter. The study protocol will specify how water and sediment will be added to the aquaria and how system will be allowed to equilibrate before organisms are introduced. The sediment will not be changed during the course of the study.

Feeding of organisms will be *ad libitum* and cleaning of tanks will occur daily. The details of how the tanks will be cleaned (particularly any measures to mitigate fiber loss) will be addressed in the study protocol.

Test Materials

Spiking material for water will be provided by the U.S Geological Survey (USGS). This material is LA ore collected from the mine site by the USGS and ground and sieved to produce material with a particle size distribution (PSD) that is generally similar to that seen in environmental media at the Libby site. Attachment A illustrates a comparison of OU3 surface water PSD to the PSD of the material that USGS has collected from the mine site.

The water used for the amphibian study will be de-chlorinated laboratory water. This will be used for both the control water and as the diluent for preparing all aqueous chemical solutions used in this study. De-chlorination will be performed by the testing laboratory by passing laboratory water through three filters: 1) a 10 inch Big Blue™ pre-treatment filter (5.0 µm) to remove solids; 2) a 3.6 cubic foot activated virgin carbon treatment filter to remove chlorine, ammonia, and higher molecular weight organics; and 3) a 5.0 µm post-treatment filter to remove any carbon particles from the carbon treatment phase.

In this study, a single water dilution will be evaluated (10 BFL), along with a laboratory control. The approach for preparation of a stock suspension to prepare this dilution will be described in the detailed protocol prepared by the toxicity testing laboratory. However, it is anticipated that EPA will provide a number of sealed ampoules of LA in water than may be used to prepare the exposure fluid with minimal effort.

Four "lots" of sediment will be collected from the Tailings Impoundment within OU3 since previous RI sampling results indicate sediment concentrations of greater than or equal to 2% at this location. Sediment samples will be collected from approximately the top four inches of the impoundment sediment in accordance with OU3 Standard Operating Procedure (SOP) 5 Revision 3, the SOP for sediment sampling (Attachment B), and documented in accordance with Libby SOP 9 Revision 5 (included as part of SOP 5 Rev. 3). Each "lot" of sediment will be a

sufficient quantity for the complete amphibian toxicity testing investigation (approximately 15 liters) plus 5 additional liters for pre-testing for LA, for a total of 20 liters per "lot". Five replicate 1-liter samples from each "lot" will be analyzed for LA by PLM-VE testing to confirm the concentration of LA in each "lot" of sediment.

The 20 replicate 1-liter samples will be sent for preparation to:

Todd Burgessor
CDM Soil Processing Facility
2714 Walnut St.
Denver CO 80205

Samples will be prepared in accordance with ISSI-LIBBY-01 Revision 10 (Attachment C). In brief, the raw sediment sample is dried and then split into two aliquots. One aliquot is placed into archive, and the other aliquot is sieved into coarse (> ¼ inch) and fine fractions. The fine fraction is ground to reduce particles to a diameter of 250 um or less and this fine-ground portion is split into 4 aliquots.

The analytical laboratory will be specified in the study protocol. Each sediment sample will be analyzed for LA in accordance with Libby site-specific SOPs. The coarse fraction (if any) will be examined using stereomicroscopy, and any particles of LA will be removed and weighed in accordance with SRC-LIBBY-01 Revision 2 (Attachment C). One of the fine ground fraction aliquots will be analyzed by polarized light microscopy (PLM) using the visual area estimation method (PLM-VE) in accordance with SRC-LIBBY-03 Revision 2 (Attachment C). Mass fraction estimates and optical property details will be recorded on the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheets.

The sediment "lot" having the consistently highest LA concentration will be used for the amphibian toxicity testing.

The source for the reference sediment will be a pond local to the testing laboratory that is known to be clean, free from asbestos, and matched as closely as possible to OU3 sediment in terms of particle size distribution and total organic carbon. The synthetic sediment will be clean sand that the laboratory used for control purposes.

Test Species and Life Stage

Based on on-site observations and data available for Lincoln County, Montana, there are four frog and toad species identified as potentially occurring at OU3 including the western toad (*Bufo boreas*), the Columbia spotted frog (*Rana luteiventris*), the Rocky Mountain tailed frog (*Ascaphus montanus*) and the Pacific treefrog (*Pseudacris regilla*). However, none of these species are available from commercial sources for use in toxicity testing, and the collection of egg masses on-site is not considered feasible. Several ranid species are available commercially for use in toxicity testing, including the Southern leopard frog (*Rana sphenocephala*), the Northern leopard frog (*Rana pipiens*), the green frog (*Rana clamitans*), and the wood frog (*Rana*

sylvatica). The test species will be one of these Ranid species, because they are good surrogates for the Columbia spotted frog (*R. luteiventris*) present on the site and are also surrogates for the other North American species present on-site. *Rana pipiens* will be the preferred test species. If *Rana pipiens* eggs are not available then the following will be used in order of preference: *Rana sphenocephala* and *Rana clamitans*. Bullfrogs (*Rana catesbeiana*) will not be used because they are considered to be more tolerant in comparison to the other ranid species. The source of the test species will be identified in the study protocol.

Egg masses will be cultured and larva tracked until at least 75% of the control animals complete metamorphosis (Gosner stage 46, see Figure 4-9). This is expected to require approximately 45-60 days.

Measurements Performed During the Study

Water Quality Measurements

Aliquots of water will be removed from each of the four LA-spiked replicate chambers and from one of the reference sediment replicate chambers (selected at random) at beginning and end of each static renewal. Each aliquot will consist of 5-10 mL withdrawn from the middle of the water column, being careful not to disturb the sediment. All water samples will be analyzed by PCM (Libby OU3 Water PCM Analysis Mod 1 – Attachment C) to provide fast turn-around results to ensure that fiber loss is not occurring.

Temperature, pH, and dissolved oxygen (DO) in water will be measured three times per week. Ammonia-nitrogen will be measured once per week.

Biological Measurements Obtained During the Study

All animals will be observed daily. Data that will be recorded daily shall include:

- hatching success
- survival counts
- developmental stage and metamorph counts
- other observations on occurrence of malformations or other abnormalities

All animals will be weighed at metamorphosis. Study log sheets will be provided in the study protocol. It should be noted that if survival becomes $\leq$ 80% in the control groups, the study will be terminated until the cause of mortality in the controls is determined.

Biological Measurements at Study Termination

Study termination is defined as the time at which 75% of the controls have completed metamorphosis. All animals will be anesthetized at study termination, digitally photographed, weighed, and examined for external abnormalities. Growth will be assessed by length (whole

body and snout-vent). A blood sample will be withdrawn and stored as plasma for potential future analysis. Metamorphosed specimens that die prior to the final stage will undergo the same procedures.

The body cavity will then be opened and all major internal organs will be inspected for developmental stage and appearance. Necropsy observations will be recorded and a second set of digital photos taken. Necropsy will also include collection of head tissue (thyroid histology), tail tissue (thyroid hormone receptor analysis), and blood (thyroid hormone) The head will be separated from the body, and both will be preserved for potential future examination of organs.

Analytical Requirements

The approach for water sample preparation and analysis will be described in the detailed protocol prepared by the toxicity testing laboratory. The protocol will be based on the results of analytical pilot studies being performed by EPA. The details of these pilot studies are described in "*Libby OU3 Pilot Study Design, Study 1A, Effect of Treatment on LA Fiber Integrity (October 20, 2010)*" and "*Libby OU3 Pilot Study Design, Study 3A, Evaluation of Field Filtration of Water Samples (October 20, 2010)*".

It is anticipated that all or a selected subset of water samples will be analyzed by PCM utilizing the PCM counting and stopping rules specified in Libby OU3 Water PCM Analysis Mod 1 (Attachment C). Selected filters (from the first, third, and final Monday of the test) will also be analyzed by TEM to confirm the results. The details of the TEM analysis method will be specified after the water analytical pilot studies are completed and will be described in the detailed protocol prepared by the toxicity testing laboratory.

Quality Control for PCM

Two types of laboratory-based QC analyses will be prepared for the PCM water samples, as follows:

Lab Blank - This is a filter through which is filtered 2.0 mL of dechlorinated laboratory water. The purpose is to evaluate whether the laboratory water used in the study contains any fibers. One laboratory blank will be prepared and analyzed each day that PCM analyses are performed. The acceptance criterion for this type of QC sample is that the number of PCM fibers in an examination of 100 fields-of-view (FOVs) does not exceed 7. If a lab blank with more than 7 fibers per 100 FOVs occurs, the laboratory should cease analytical activities until the source of contamination is identified and corrected.

Blind Recounts - A total of 5% of all PCM slides will be submitted for blind recounts. In this procedure, a slide that has been analyzed is re-labeled by a person other than the original analyst and re-submitted for a second analysis. The acceptance criterion for this type of QC sample is that no more than 5% of the re-analysis pairs are statistically different from each other.

Quality Control for TEM

Two types of laboratory-based QC analyses will be prepared for the TEM water samples, as follows:

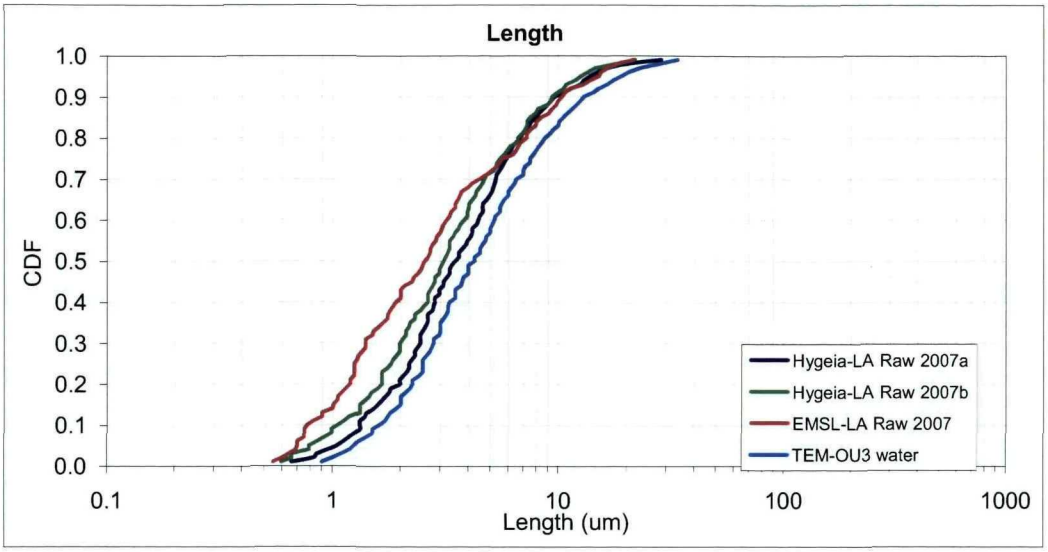
> *Lab Blank* - This is an analysis of a TEM grid that is prepared from a new, unused filter in the laboratory and is analyzed using the same procedure as used for field blank samples. One lab blank should be prepared and analyzed along with the water samples selected for TEM analysis. The acceptance criterion for this type of QC sample is that no asbestos structures should be observed in an examination of 10 GOs. If one or more asbestos structures are observed, the laboratory should cease analytical activities until the source of contamination is identified and corrected.

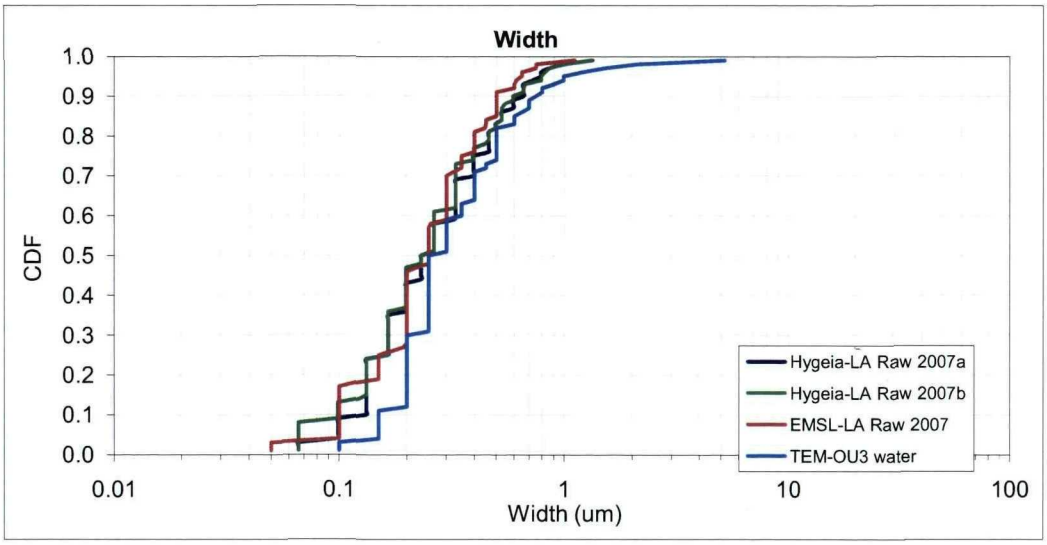
Recounts - A recount is an analysis where TEM grid openings are re-examined after the initial examination. A *Recount Different* (RD) describes a re-examination by a different microscopist within the same laboratory than who performed the initial examination. A total of two samples will be selected by SRC for Recount Different (RD) analysis after the results of the original sample analyses have become available. The most recent version of laboratory modification LB-000029 (see Attachment C) summarizes the acceptance criteria for these Recount Different analyses.

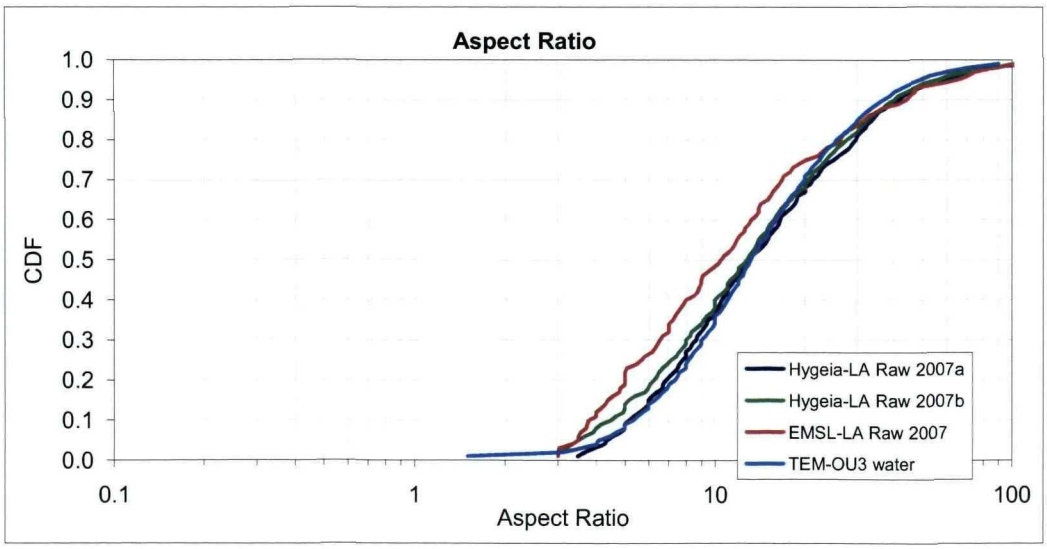
ATTACHMENT A

LA Particle Size Distributions for LA Raw 2007 Material and OU3 Water

Particle Size Distributions of LA Particles* - Hygeia LA Raw 2007a (N = 1020), Hygeia LA Raw 2007b (N = 999), EMSL LA Raw 2007 (N = 196), OU3 Water (N = 4,330)







*Raw material results have been normalized so that only LA structures with a length greater than 0.5 and aspect ratio greater to or equal than 3 have been included. Structures have not been excluded for crossing grid bars and lengths have not been doubled in these cases.

ATTACHMENT B

Sediment Sampling and Field Documentation
Standard Operating Procedures

OU3 SOP 5 (Rev. 3) – Sediment Sampling
OU3 SOP 9 (Rev. 5) – Field Documentation

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Date: November 19, 2010 OU3 SOP 5 (Rev. 3)

Title: SEDIMENT SAMPLING

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager		11/19/10
SOP Author		11/19/10

Revision Number	Date	Reason for Revision
3	November 19, 2010	Incorporates sediment collection with suction assisted sediment sampling device (SASSD)

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) describes methods and equipment commonly used for collecting environmental samples of sediment. The information presented in this SOP is applicable to the collection of representative sediment samples. Analysis of sediment may be biological, chemical, or physical in nature and may be used to determine the following:

- toxicity
- biological availability and effects of contaminants
- benthic biota
- extent and magnitude of contamination
- contaminant migration pathway and potential source
- fate of contaminants
- grain size distribution

The methodologies discussed in this SOP are applicable to the sampling of sediment in both flowing and standing water. For the purposes of this procedure, sediments are those mineral and organic materials situated beneath an aqueous layer. The water may be static, as in lakes, ponds, and impoundments; or flowing, as in rivers and streams. The document focuses on methods and equipment that are readily available and typically applied in collecting sediment samples. It is not intended to provide an all-inclusive discussion of sample collection methods. Specific sampling problems may require the adaptation of existing equipment or the design of new equipment. Such innovations shall be clearly described in the project-specific sampling plan and approved by the Project Manager.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in sediment sampling must follow health and safety protocols described in the health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of

lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR, 2006).

Fabrication of an SASSD requires the use of polyvinyl chloride (PVC) cement on non-threaded connections. PVC cement contains the volatile organic compounds tetrahydrofuran, methyl ethyl ketone and cyclohexanone. A Material Safety Data Sheet for typical PVC cement is provided as Attachment A. PVC cement should be used only in areas with adequate ventilation that are free of ignition sources, and all manufacturer's directions and precautions must be followed. Because samples collected with an SASSD may be analyzed for one or more of the volatile constituents contained in PVC cement, the fabricated device must be allowed to thoroughly air-dry and cure for at least 24 hours prior to use.

Operation of an SASSD typically is done from a boat, thus water-safety procedures must be employed and approved personal floatation devices must be worn by all onboard sampling personnel.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information will be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel will always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects site-specific field sampling program with input from other key project staff, and applicable oversight agencies.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Sampling Technician (or other designated personnel): Assists the FTL and/or geologist, hydrogeologist, or engineer in the implementation of tasks. Performs the actual sample collection, packaging, and documentation (e.g., sample label and log sheet, chain-of-custody record, etc).

4.0 SEDIMENT SAMPLING PROCEDURES

Sediment samples may be collected using a variety of methods and equipment, depending on the depth of the aqueous layer, the portion of the sediment profile required (surface vs. subsurface), the type of sample required (disturbed vs. undisturbed), contaminants present, and sediment type. Sediment is collected from beneath an aqueous layer either directly, using a hand held device such as a shovel, trowel, or auger; or indirectly, using a remotely activated device such as an Ekman or Ponar dredge. Following collection, sediment is transferred from the sampling device to a sample container of appropriate size and construction for the analyses requested. If composite sampling techniques are employed, multiple grabs are placed into a container constructed of inert material, homogenized, and transferred to sample containers appropriate for the analyses requested.

At Libby OU3, the sampling of sediments is anticipated to occur by one of the following methods:

- Sampling with a Trowel or Scoop from Beneath a Shallow Aqueous Layer
- Sampling with a Bucket Auger, Tube Auger, or other Coring Device from Beneath a Shallow Aqueous Layer
- Sampling Sediment with a Suction-Assisted Sediment Sampling Device
- Sampling Surface Sediment with an Ekman or Ponar Dredge from Beneath a Shallow or Deep Aqueous Layer

4.1 Equipment

The selection of sampling equipment listed depends on the site conditions and sample type required.

- Spade, Shovel, Trowel or Scoop: used for collecting sediment samples from shallow (wadable) locations.
- Bucket Auger or Tube Auger: used for collecting sediment samples from shallow (wadable) locations.
- Ekman or Ponar dredge: used for collecting sediment samples from lakes and ponds.
- Nylon rope or steel cable: for raising and lowering the dredge
- Collection containers: 8-oz and one-quart wide mouth glass jars with Teflon lined lids.
- Gloves: for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.
- Field clothing and Personal Protective Equipment as specified in the Health and Safety Plan.
- Sampling flags: used for identifying sediment sampling locations.
- Field notebook: a bound book used to record progress of sampling effort and record any problems and field observations during sampling.
- Three-ring binder book: to store necessary forms used to record and track samples collected at the site.
- Permanent marking pen: used to mark soil boring tubes and for documentation of field logbooks and data sheets.
- Stainless Steel lab spoon or equivalent: used for homogenizing sediment samples that will not be used for VOCs analysis or toxicity testing
- Stainless Steel Buckets: used for compositing samples that will not be used for VOCs analysis or toxicity testing. Must have 10-12 liter capacity.
- Trash Bag: used to dispose of gloves and any other non-hazardous waste generated during sampling
- Decontamination supplies/equipment

4.2 General Sampling Procedures

Collect surface water samples according to SOP-3 prior to collecting sediment samples. Stream sediment samples will be composite samples comprised of five subsamples collected from the

surface to a depth of 4 inches, which is the most biologically active sediment zone. The five subsamples will be collected from random locations along the creek channel within 200 feet of the specified location. No sediments will be collected from overbank areas, unless specified in the FSP.

For collecting sediment samples, the procedures outlined below shall be followed.

1. Don appropriate health and safety equipment.

2. Setup clean plastic sheeting in area for processing samples.

3. Collect sediment samples using the appropriate decontaminated equipment (described in Sections 6.2, 6.3, and 6.4) from inundated areas beginning at the most downstream location (i.e., no sediments will be collected from overbank areas).

4. Composite the five subsamples with sufficient volume to meet requirements for testing (as specified for each sampling location in the workplan) into a homogenization container (usually a stainless steel bucket) and homogenize by stirring.

5. Carefully remove twigs, rocks, leaves and other undesirable debris not considered part of the sample. Distribute the homogenized sediment into sampling containers (type, number and size specified in workplan).

6. Label the sampling containers with the Index ID, sample location, and sample analysis information in accordance with the procedures in SOP No. 9. Place in cooler on ice for storage and shipment (refer to SOP-8 for sample handling and shipping information).

7. Complete the appropriate sediment Field Sample Data Sheet (FSDS) form to document the station and sample details (see SOP No. 9, Attachment B). Document sediment characteristics, sample location as well as any changes to this SOP in the field logbook.

8. Locate the sample using a site map or GPS according to SOP-11.

For duplicates, a second sediment composite sample will be collected (Steps 3-8 above) from the sampling reach and placed into sample containers.

4.3 Equipment Specific Sampling Procedures

4.3.1 Sampling with a Trowel or Scoop from Beneath a Shallow Aqueous Layer

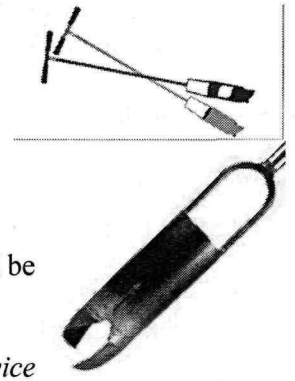
Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with tools such as spades, shovels, trowels, and scoops. Although this method can be used to collect both unconsolidated/consolidated sediment, it is limited somewhat by the depth and movement

of the aqueous layer. Deep and rapidly flowing water render this method less accurate than others discussed below. However, representative samples can be collected with this procedure in shallow sluggish water provided care is demonstrated by the sample team member. A stainless steel or plastic sampling implement will suffice in most applications. Care should be exercised to avoid the use of devices plated with chrome or other materials; plating is particularly common with garden trowels. The following procedure will be used to collect sediment with a scoop, shovel, or trowel:

1. Using a decontaminated sampling implement, remove the desired thickness and volume of sediment from the sampling area.

2. Transfer the sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.

3. Surface water should be decanted from the sample or homogenization container prior to sealing or transfer; care should be taken to retain the fine sediment fraction during this procedure.



4.3.2 Sampling with a Bucket Auger, Tube Auger, or other Coring Device from Beneath a Shallow Aqueous Layer

Collection of surface sediment from beneath a shallow aqueous layer can be accomplished with a system consisting of bucket auger or tube auger, a series of extensions, and a "T" handle. The use of additional extensions in conjunction with a bucket auger can increase the depth of water from which sediment can be collected from 24 inches to 10 feet or more. However, sample handling and manipulation increases in difficulty with increasing depth of water. The bucket auger or tube auger is driven into the sediment and used to extract a core. The various depths represented by the core are homogenized or a subsample of this core is taken from the appropriate depth. The following procedure will be used to collect sediment samples with a bucket auger or tube auger:

1. An acetate core may be inserted into the bucket auger or tube auger prior to sampling if characteristics of the sediments or waterbody warrant. By using this technique, an intact core can be extracted.

2. Attach the auger head to the required length of extensions, then attach the "T" handle to the upper extension.

3. Clear the area to be sampled of any surface debris.

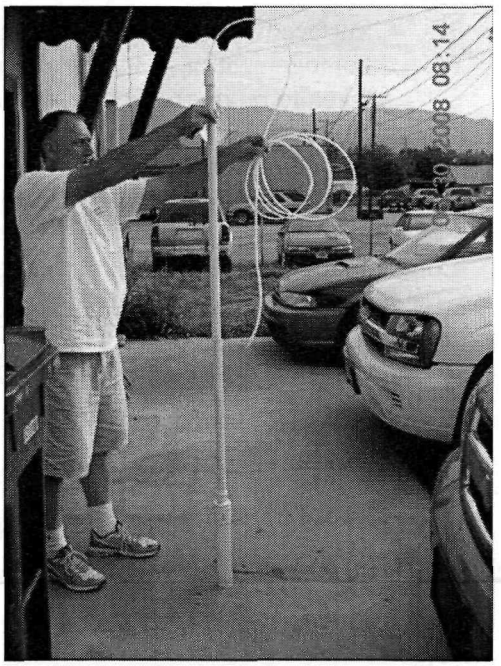
4. Insert the bucket auger or tube auger into the sediment at a 0° to 20° angle from vertical. This orientation minimizes spillage of the sample from the sampler upon extraction from the sediment and water.

5. Rotate the auger to cut a core of sediment.

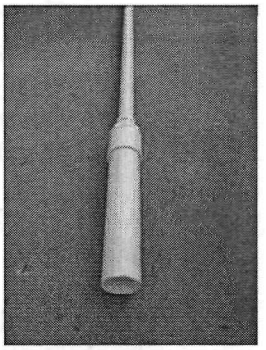
6. Slowly withdraw the auger; if using a tube auger, make sure that the slot is facing upward.

7. Transfer the sample or a specified aliquot of sample into an appropriate sample or homogenization container. Ensure that non-dedicated containers have been adequately decontaminated.

4.3.3 Sampling Sediment with a Suction-Assisted Sediment Sampling Device



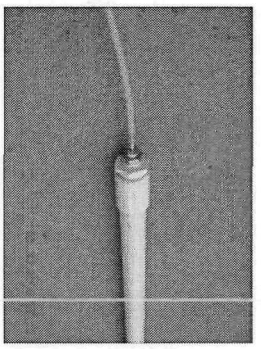
An Suction-Assisted Sediment Sampling Device (SASSD) (see picture) consists of three basic parts: 1.) a sampling head, which consists of a 12-inch length of 2-inch diameter Schedule 40 PVC pipe, open at the bottom and solvent-welded at the top to a 2-inch slip to 1-inch female threaded reducing adapter (see Figures 2 and 3); 2.) an extension tube (five, ten or 15 feet long) of 1-inch diameter Schedule 40 PVC pipe, solvent-welded to a 1-inch male threaded connector on the bottom and a 1-inch to 3/8-inch brass reducing bushing to which a 3/8-inch to 1/8-inch barbed nipple is attached to a length of 1/8-inch polyethylene tubing attached to the barbed brass nipple. The polyethylene tubing must be of sufficient length (five, ten or 15 feet, depending on the water depth and length of extension tube being used) to allow the sampling head of the SASSD to be fully lifted above the water while still attached to the peristaltic pump, which remains onboard the boat. The polyethylene tubing is attached to a two-foot length of ¼-inch silicone rubber tubing that is fitted into the peristaltic pump drive cam casing.



A positive-displacement pump (e.g., a GeoPump II®, manufactured by GeoTech, Inc.) is used to operate the SASSD. Fluid (in this application, air) is contained within a flexible tube (typically silicone rubber) fitted inside a circular pump drive cam casing. A rotor with a number of cams (typically three) attached to its external circumference compresses the flexible tubing. As the rotor turns, the part of tube under compression closes, thus forcing the air to be pumped through the tube. As the tube opens to its uncompressed state after the cam passes, air flow is induced through the tubing. The GeoPump II is reversible (i.e., air can be driven in either direction, under either negative or positive pressure). Attachment C is product literature and specifications for the GeoPump II used at OU3.

The SASSD is operated using the following steps:

1. The water depth at the location to be sampled with an SASSD should be sounded with a weighted measuring tape or electronic "fish-finder" so that an extension tube of adequate length can be selected. The shortest extension tube necessary to reach the pond bottom yet still extend above the water level should be selected and attached to the sampling head. Teflon tape may be used on the threads if they become worn and cause a loss of suction.



2. With the tubing attached and the peristaltic pump off, lower the SASSD vertically into the water until the lower end of the sampling head contacts the bottom of the pond. Leaving the pump off prevents water from entering the sampling head and extension tube and ensures that less water is collected with the sediment sample.

3. Once the sampling head is firmly on the bottom of the pond floor, push the assembly one foot into the sediment. Resistance should be felt when the sampling head is full. Turn the peristaltic pump on, with the speed set to medium (approximately 150 rpm on the GeoPump II), so that suction is applied through the pump tubing and extension tube. As a general guide, the times to produce suction sufficient to retain the sediment sample and overcome the suction of the bottom sediments surrounding the sampling head (yet not pull water or sediment up into the extension tube or pump tubing) are as follows:

Extension Tube Length	Pumping Time
5-foot	20 seconds
10-foot	45 seconds
15-foot	75 seconds

Pumping times for extension tubes of various lengths will vary according to pump type, pump speed, sediment characteristics and pump battery condition. Experimentation may be necessary to yield optimum results. Care must be taken to ensure the suction is no more than that needed to retain the sediment in the sampling head, so that little or no water or sediment is pulled up the extension tube or into the pump tubing.

4. At the conclusion of the pumping period, grasp the upper portion of the extension tube and firmly, steadily pull the sampling head free from the pond bottom. A slight side-to-side motion sometimes is helpful in breaking the bottom suction and overcoming the friction of the bottom sediments that surround the sampling head. Once the sampling head is free, quickly raise the sampling head out of the water, place the end of the sampling head in a decontaminated stainless steel bowl, and reverse the flow direction on the peristaltic pump controller. Positive pressure from the pump will extrude the sediment core into the bowl (see Figure 4). Take care to note that the top end of the core is extruded last. Unless otherwise noted, only the upper four inches of the sediment core is retained for OU3 pond sediment samples; the balance of the core is set aside for later disposal on the bank of the pond or at the Amphitheater disposal site. Repeat the sampling procedure until an adequate volume of four-inch core segments has been collected to fill the required sample containers.

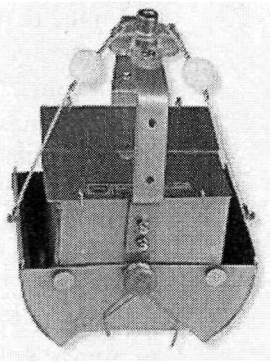
   The sample shall then be handled and distributed among the analytical bottles as described earlier in Section 4.2.

The SASSD tubing should be inspected periodically to ensure that kinks, tears or pinholes do not develop. The threaded connection between the sampling head and the extension tube should be inspected and cleaned during decontamination to ensure a tight seal is maintained. The peristaltic pump battery should be fully charged at the beginning of each day of sampling, and a spare fully-charged battery should be on-hand. Spare polyethylene and silicone rubber tubing should be readily available for replacement of worn or damaged tubing. All maintenance activities must be recorded in the field logbook or on field forms following SOP No. 9-Field Documentation.

The inner and outer surfaces of the sampling head shall be decontaminated with Alconox, a bottle brush and distilled water rinse prior to initial use and between use at each sampling station. Decontamination is not necessary between multiple sampling runs at a particular sampling station. The extension tube and pump tubing should be inspected after each sampling run to ensure that water or sediment have not been pulled up into them during sample collection under suction. If water or sediment have entered the extension tube or pump tubing, these parts shall be decontaminated by using Alconox and distilled water rinse (or in the case of pump tubing, the contaminated tubing may be discarded and replaced with new tubing). All

decontamination fluids shall be contained for later disposal at the Amphitheater, so as to not affect the quality of subsequent samples. Decontamination procedures are presented in SOP No. 7-Equipment Decontamination.

The functioning of an SASSD will be affected by loss of suction caused by kinks, holes or tears in the tubing, debris in threaded joints, or malfunctioning of the peristaltic pump. The quality of a sediment sample collected by an SASSD may also be affected by inadequate suction due to a thick layer of brittle organic detritus (e.g., twigs, bark), mineral fragments or other debris, although such a condition was not encountered during the initial round of SASSD sampling of ponds at OU3 in July, 2008. If such conditions are encountered, the sampling location should be abandoned, the equipment shall be decontaminated, and a nearby alternate sampling location should be selected.



4.3.4 Sampling Surface Sediment with an Ekman or Ponar Dredge from Beneath a Shallow or Deep Aqueous Layer

Collection of surface sediment can be accomplished with a system consisting of a remotely activated device (dredge) and a deployment system. This technique consists of lowering a sampling device (dredge) to the surface of the sediment by use of a rope, cable, or extended handle. The mechanism is activated, and the device entraps sediment in spring loaded or lever operated jaws. An Ekman dredge is a lightweight sediment sampling device with spring activated jaws. It is used to collect moderately consolidated, fine textured sediment. The following procedure will be used for collecting sediment with an Ekman dredge:

1. Attach a sturdy nylon rope or stainless steel cable through the hole on the top of the bracket, or secure the extension handle to the bracket with machine bolts.

2. Attach springs to both sides of the jaws. Fix the jaws so that they are in open position by placing trip cables over the release studs. Ensure that the hinged doors on the dredge top are free to open.

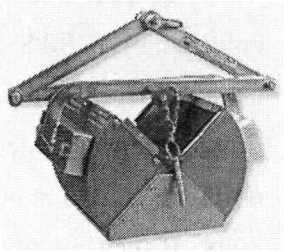
3. Lower the sampler to a point 4 to 6 inches above the sediment surface.

4. Drop the sampler to the sediment.

5. Trigger the jaw release mechanism by lowering a messenger down the line, or by depressing the button on the upper end of the extension handle.

6. Raise the sampler and slowly decant any free liquid through the top of the sampler. Care should be taken to retain the fine sediment fraction during this procedure.

7. Open the dredge jaws and transfer the sample into a stainless steel, plastic or other container by depressing the button on the upper end of the should be taken to retain the fine sediment appropriate composition (e.g., Teflon) container. Ensure that non-dedicated containers have been adequately decontaminated. If necessary, continue to collect additional sediment grabs until sufficient material has been secured to fulfill analytical requirements. Thoroughly homogenize and then transfer sediment to sample containers appropriate for the analyses requested. Samples for volatile organic analysis must be collected directly from the bucket before homogenization to minimize volatilization of contaminants.



A Ponar dredge is a heavyweight sediment sampling device with weighted jaws that are lever or spring activated. It is used to collect consolidated fine to coarse textured sediment. The following procedure will be used for collecting sediment with a Ponar dredge:

1. Attach a sturdy nylon rope or steel cable to the ring provided on top of the dredge.

2. Arrange the Ponar dredge with the jaws in the open position, setting the trip bar so the sampler remains open when lifted from the top. If the dredge is so equipped, place the spring loaded pin into the aligned holes in the trip bar.

3. Slowly lower the sampler to a point approximately two inches above the sediment.

4. Drop the sampler to the sediment. Slack on the line will release the trip bar or spring loaded pin; pull up sharply on the line closing the dredge.

5. Raise the dredge to the surface and slowly decant any free liquid through the screens on top of the dredge. Care should be taken to retain the fine sediment fraction during this operation.

6. Open the dredge and transfer the sediment to a stainless steel, plastic or other appropriate composition (e.g., Teflon) container. Ensure that non-dedicated containers have been adequately decontaminated. If necessary, continue to collect additional sediment until sufficient material has been secured to fulfill analytical requirements. Thoroughly homogenize the sediment and then transfer sediment to sample containers appropriate for the analyses requested. Samples for volatile organic analysis must be collected directly from the bucket before homogenization to minimize volatilization of contaminants.

5.0 QUALITY ASSURANCE AND QUALITY CONTROL

Field splits, field blanks, equipment rinsates, and matrix spike samples will be collected at the frequencies documented in the field sampling plan. Calibration checks will be performed at least once prior to and at least once following each day of instrument use in the field and the results documented in the field log book. All sampling data must be documented in the field logbooks and/or field forms, including rationales deviations from this SOP. The Field Team Leader or designated QA reviewer will check and verify that field documentation has been completed per this procedure and other procedures referenced herein. All equipment must be operated according to the manufacturer's specifications, including calibration and maintenance.

6.0 DECONTAMINATION

All equipment used in the sampling process shall be decontaminated prior to field use and between sample locations. Decontamination procedures are presented in SOP-7. Personnel shall don appropriate personal protective equipment as specified in the health and safety plan. Any investigation-derived waste generated in the sampling process shall be managed in accordance with the procedures outlined in SOP-12.

7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

Barth, D.S., and B.J. Mason. 1984. Soil sampling quality assurance user's guide. USEPA_600/4_84_043.

de Vera, E.R., B.P. Simmons, R.D. Steohen, and D.L. Storm. 1980. Samplers and sampling procedures for hazardous waste streams. EPA_600/2_80_018.

Mason, B.J. 1983. Preparation of soil sampling protocol: techniques and strategies. EPA_600/4_83_020.

USEPA. 1984. Characterization of hazardous waste_ a methods manual: Volume II: Available sampling methods, second edition. EPA_600/4_84_076.

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ATTACHMENT A

Material Safety Data Sheet for PVC Cement Components

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IPS
WELD-ON

MATERIAL SAFETY DATA SHEET

Date Revised: FEB 2005
Supersedes: OCT 2004

Information on this form is furnished solely for the purpose of compliance with the Occupational Safety and Health Act and shall not be used for any other purpose. IPS Corporation urges the customers receiving this Material Safety Data Sheet to study it carefully to become aware of the hazards, if any, of the product involved. In the interest of safety, you should notify your employees, agents and contractors of the information on this sheet.

SECTION I

MANUFACTURER'S NAME
IPS Corporation
ADDRESS
17109 S. Main St., P.O. Box 379, Gardena, CA. 90248

Transportation Emergencies:
CHEMTREC: (800) 424-9300
Medical Emergencies:
3 E COMPANY (24 Hour No.) (800) 451-8346
Business: (310) 898-3300

CHEMICAL NAME and FAMILY
Solvent Cement for PVC Plastic Pipe
Mixture of PVC Resin and Organic Solvents

TRADE NAME:
WELD-ON 702, 704, 705, 707, 710, 711, 717, 719 and 721 for PVC Plastic Pipe
FORMULA: Proprietary

SECTION II - HAZARDOUS INGREDIENTS

None of the ingredients below are listed as carcinogens by IARC, NTP or OSHA	CAS#	APPROX %	ACGIH-TLV	ACGIH-STEL	OSHA-PEL	OSHA-STEL	DUPONT (A) AEL	DUPONT (B) STEL
Polyvinyl Chloride Resin (PVC)	NON/HAZ		N/A		N/A			
Tetrahydrofuran (THF)**	109-99-9	25 - 70	200 PPM	250 PPM	200 PPM	250 PPM	50 PPM	75 PPM
Methyl Ethyl Ketone (MEK)	78-93-3	5 - 40*	200 PPM	300 PPM	200 PPM	300 PPM		
Cyclohexanone	108-94-1	1 - 15	20 PPM Skin	50 PPM	50 PPM Skin			

All of the constituents of Weld-On adhesive products are listed on the TSCA inventory of chemical substances maintained by the US EPA, or are exempt from that listing.

* Title III Section 313 Supplier Notification: This product contains toxic chemicals subject to the reporting requirements of Section 313 of the Emergency Planning and Community Right-to-Know Act of 1986 and of 40CFR372. This information must be included in all MSDS's that are copied and distributed for this material.

(A) Dupont and BASF Mfg's Acceptable Exposure Limit (AEL) guidelines for 8 hour and 12 hour TWA, (B) Dupont/BASF recommended STEL for 15 minute TWA.

**Information found in a report from the National Toxicology Program (NTP) on an inhalation study in rats and mice suggests that Tetrahydrofuran (THF) can cause tumors in animals. In the study the rats and mice were exposed to THF vapor levels up to 1800 PPM for two years (their lifetime), 6 hours/day, 5 days/week. Test results showed evidence of liver tumors in female mice and kidney tumors in male rats. No evidence of tumors was seen in female rats and male mice. There is no data linking Tetrahydrofuran exposure with cancer in humans.

BULK SHIPPING INFORMATION / CONTAINERS LARGER THAN ONE LITER

DOT Shipping Name:	Adhesive
DOT Hazard Class:	3
Identification Number:	UN 1133
Packaging Group:	II
Label Required:	Flammable Liquid

SHIPPING INFORMATION FOR CONTAINERS LESS THAN ONE LITER

DOT Shipping Name:	Consumer Commodity
DOT Hazard Class:	ORM-D

SPECIAL HAZARD DESIGNATIONS

	HMIS	NFPA	HAZARD RATING
HEALTH:	2	2	0 - MINIMAL
FLAMMABILITY:	3	3	1 - SLIGHT
REACTIVITY:	0	1	2 - MODERATE
PROTECTIVE			3 - SERIOUS
EQUIPMENT:	B - H		4 - SEVERE

B = Eye, Hand/Skin (for normal solvent-welding, small spill, clean-up activities)
H = Eye, Hand/Skin, Respiratory Protection and Impermeable Apron (splash/ immersion risks)

SECTION III - PHYSICAL DATA

APPEARANCE	ODOR	BOILING POINT (°F/°C)	FREEZING POINT
704 - clear or gray, medium syrupy liquid; 705 - gray, clear or white, medium syrupy liquid; 702, 707 - clear, medium syrupy liquid; 710 - clear, thin syrupy liquid; 711 - white or opaque gray, heavy syrupy liquid; 717 - opaque gray, clear or white heavy syrupy liquid; 719 - clear, gray, green or white, paste-like; 721 - blue, medium syrupy liquid	Ethereal (Threshold = 2-50 PPM)	151°F (67°C) Based on THF	-163°F (-108.5°C)

SPECIFIC GRAVITY @ 73°F ± 3.6° (23°C ± 2°)	VAPOR PRESSURE (mm Hg.)	PERCENT VOLATILE BY VOLUME (%)
Variable by product ranging from 0.900 to 0.981 ± 0.040	143 mm Hg. based on first boiling component, THF @ 68°F (20°C)	Approx: 80 - 90 %

VAPOR DENSITY (Air = 1)	EVAPORATION RATE (BUAC = 1)	SOLUBILITY IN WATER
2.49	> 1.0	Solvent portion completely soluble in water. Resin portion separates out.

VOC STATEMENT: VOC as manufactured: 850 Grams/Liter (g/l). Maximum VOC emission when applied and tested per SCAQMD Rule 1168, Test Method 316A: 600 g/l.

SECTION IV - FIRE AND EXPLOSION HAZARD DATA

FLASH POINT	FLAMMABLE LIMITS (PERCENT BY VOLUME)	LEL	UEL
-4°F (-20°C) T.C.C. Based on THF		2.0	11.8

FIRE EXTINGUISHING MEDIA
Ansul "Purple K" potassium bicarbonate dry chemical, any appropriately sized ABC dry chemical, carbon dioxide or foam extinguisher can be used for small fires. Use of a water fog by trained personnel can extinguish small/large fires.

SPECIAL FIRE FIGHTING PROCEDURES
Evacuate enclosed areas. Stay upwind. Close quarters or confined spaces require self-contained breathing apparatus, positive pressure mask or airline mask. Use of a water fog by trained personnel can extinguish small/large fires and avoid water flow or water streams/spray distributing burning material or contaminated water over a large area or into sewers or storm drains. Use water spray to cool containers, to flush spills from source of ignition and to disperse vapors.

UNUSUAL FIRE AND EXPLOSION HAZARDS
Fire hazard because of low flash point and high volatility. Vapors are heavier than air and may travel to source(s) of ignition at or near ground or lower level(s) and may flash back.

ff-d

SECTION V - HEALTH HAZARD DATA

PRIMARY ROUTES OF ENTRY: ___X___ Inhalation ___X___ Skin Contact _____ Eye Contact _____ Ingestion

EFFECT OF OVEREXPOSURE

ACUTE:

Inhalation: Severe overexposure may result in nausea, dizziness, headache. Can cause drowsiness, irritation of eyes and nasal passages.

Skin Contact: Skin irritant. Liquid contact may remove natural skin oils resulting in skin irritation. Dermatitis may occur with prolonged contact.

Skin Absorption: Prolonged or widespread exposure may result in the absorption of harmful amounts of material.

Eye Contact: Overexposure may result in severe eye injury with corneal or conjuctival inflammation on contact with the liquid. Vapors slightly uncomfortable.

Ingestion: Moderately toxic. May cause nausea, vomiting, diarrhea. May cause mental sluggishness.

CHRONIC: Symptoms of respiratory tract irritation and damage to respiratory epithelium were reported in rats exposed to 5000 ppm THF for 90 days. Elevation of SGPT suggests a disturbance in liver function. The NOEL was reported to be 200 ppm.

REPRODUCTIVE EFFECTS	TERATOGENICITY	MUTAGENICITY	EMBRYOTOXICITY	SENSITIZATION TO PRODUCT	SYNERGISTIC PRODUCTS
N. AP.	N. AP.	N. AP.	N. AP.	N. AP.	N. AV.

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Individuals with pre-existing diseases of the eyes, skin or respiratory system may have increased susceptibility to the toxicity of excessive exposures.

EMERGENCY AND FIRST AID PROCEDURES

Inhalation: If overcome by vapors, remove to fresh air and if breathing stopped, give artificial respiration. If breathing is difficult, give oxygen. Call physician.

Eye Contact: Flush eyes with plenty of water for 15 minutes and call a physician.

Skin Contact: Remove contaminated clothing and shoes. Wash skin with plenty of soap and water for at least 15 minutes. If irritation develops, get medical attention.

Ingestion: Give 1 or 2 glasses of water or milk. Do not induce vomiting. Call physician or poison control center immediately.

SECTION VI - REACTIVITY

STABILITY	UNSTABLE		CONDITIONS TO AVOID
	STABLE	X	Keep away from heat, sparks, open flame and other sources of ignition.

INCOMPATIBILITY

(MATERIALS TO AVOID) Caustics, ammonia, inorganic acids, chlorinated compounds, strong oxidizers and isocyanates.

HAZARDOUS DECOMPOSITION PRODUCTS

When forced to burn, this product gives out carbon monoxide, carbon dioxide, hydrogen chloride and smoke.

HAZARDOUS	MAY OCCUR		CONDITIONS TO AVOID
POLYMERIZATION	WILL NOT OCCUR	X	Keep away from heat, sparks, open flame and other sources of ignition.

SECTION VII - SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IN CASE MATERIAL IS RELEASED OR SPILLED

Eliminate all ignition sources. Avoid breathing of vapors. Keep liquid out of eyes. Flush with large amount of water. Contain liquid with sand or earth. Absorb with sand or nonflammable absorbent material and transfer into steel drums for recovery or disposal. Prevent liquid from entering drains.

WASTE DISPOSAL METHOD

Follow local, State and Federal regulations. Consult disposal expert. Can be disposed of by incineration. Excessive quantities should not be permitted to enter drains. Empty containers should be air dried before disposing. Hazardous Waste Code (CA): 214.

SECTION VIII - SPECIAL PROTECTION INFORMATION

RESPIRATORY PROTECTION (Specify type)

Atmospheric levels should be maintained below established exposure limits contained in Section II. If airborne concentrations exceed those limits, use of a NIOSH approved organic vapor cartridge respirator with full face-piece is recommended. The effectiveness of an air purifying respirator is limited. Use it only for a single short-term exposure. For emergency and other conditions where short-term exposure guidelines may be exceeded, use an approved positive pressure self-contained breathing apparatus.

VENTILATION

Use only with adequate ventilation. Do not use in close quarters or confined spaces. Open doors and/or windows to ensure airflow and air changes. Use local exhaust ventilation to remove airborne contaminants from employee breathing zone and to keep contaminants below levels listed in Section II. Use only explosion-proof ventilation equipment.

PROTECTIVE GLOVES PVA coated rubber gloves for frequent dipping/immersion. Use of latex/nitrile surgical gloves or solvent resistant barrier cream should provide adequate protection when normal solvent-cement welding practices and procedures are used for solvent welding of plastic sheet/pipe joints.

EYE PROTECTION Splashproof chemical goggles, face shield, safety glasses (spectacles) with brow guards & side shields, etc. as appropriate for exposure.

OTHER PROTECTIVE EQUIPMENT AND HYGIENIC PRACTICES

Impervious apron and a source of running water to flush or wash the eyes and skin in case of contact.

SECTION IX - SPECIAL PRECAUTIONS

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORING

Store in the shade between 40°F - 110°F (5°C - 43.7°C). Keep away from heat, sparks, open flame and other sources of ignition. Avoid prolonged breathing of vapor. Use with adequate ventilation. Avoid contact with eyes, skin and clothing. Train employees on all special handling procedures before they work with this product.

OTHER PRECAUTIONS

Follow all precautionary information given on container label, product bulletins and our solvent cementing literature. All material handling equipment should be electrically grounded.

ATTACHMENT B

OU3 SOP 9 (Rev. 5) – Field Documentation

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Date: May 20, 2009

OU3 SOP 9 (Rev. 5)

Title: FIELD DOCUMENTATION

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	/RPM	5/21/09
SOP Author		5/22/09

Revision Number	Date	Reason for Revision
0	09/26/2007	--
1	10/5/2007	• Add section for "Corrections and Modifications" and Field Modification Approval form (Attachment 3) • Update Labeling section and COC (Attachment 2) to reflect non-asbestos analysis and container details • Update FSDS forms (Attachment 1) based on field team input
2	02/22/2008	• Incorporate changes to FSDS forms (Attachment 1) based on field input • Remove OU3 phase specificity in SOP text
3	05/29/2008	• Incorporate changes to FSDS forms (Attachment 1) based on field input
4	06/30/2008	• Update Attachment 1 with all OU3 FSDS forms (including those used in Phase I and Phase II) • Remove OU3 phase specificity in Attachments
5	05/20/2009	• Add FSDS form for ABS Personal Air • Add FSDS form for Small Mammal Tissue • Modified COC to change medium code to "A-Air" to accommodate both ambient and activity-based sampling (ABS) air samples • Added new media code for small mammal tissue "MT"

1.0 INTRODUCTION

This Standard Operating Procedure (SOP) is a general guidance document for the required documentation to be completed by field personnel during field investigations. This SOP is based on MWH SOP-04, Field Documentation, Revision 1.0, March 2006, modified for use at the Libby Mine Site. Documentation in the form of field logbooks, reports, and forms shall be completed for every activity in the field. Records shall be maintained on a daily basis as the work progresses. All field documentation shall be accurate and legible because it is deliverable to the client as potentially a legal document.

2.0 HEALTH AND SAFETY WARNING

All personnel engaged in soil sampling must follow health and safety protocols described in the site health and safety plan. Asbestos fibers are thin and long fibers so small that they cannot be seen by the naked eye. Asbestos fibers are easily inhaled when disturbed and when embedded in the lung tissue can cause health problems. Significant exposure to asbestos increases the risk of lung cancer, mesothelioma, asbestosis (non-cancerous lung disease), and other respiratory diseases (ATSDR 2006). All personnel engaged in soil sampling must follow health and safety protocols described in the health and safety plan.

3.0 RESPONSIBILITIES

This section presents a brief definition of field roles, and the responsibilities generally associated with them. This list is not intended to be comprehensive and often, additional personnel may be involved. Project team member information shall be included in project-specific plans (e.g., work plan, field sampling plan, quality assurance plan, etc.), and field personnel shall always consult the appropriate documents to determine project-specific roles and responsibilities. In addition, one person may serve in more than one role on any given project.

Project Manager: Selects project-specific field documentation with input from other key project staff.

Quality Control Manager: Overall management and responsibility for quality assurance and quality control (QA/QC). Selects QA/QC procedures for the sampling and analytical methods, performs project audits, and ensures that data quality objectives are fulfilled.

Field Team Leader (FTL) and/or Field Geologist, Hydrogeologist, or Engineer: Implements the sampling program, supervises other sampling personnel, and ensures compliance with SOPs and QA/QC requirements. Prepares daily logs of field activities.

Field Technician (or other designated personnel): Assists the FTL and/or field geologist, hydrogeologist, or engineer in the implementation of field tasks and field documentation.

Field Sample/Data Manager: Responsible for proper handling and shipping of all samples collected by the field crew, electronic data entry of field sample data sheet (FSDS) and chain-of-custody (COC) forms, and scanning/posting of field documentation PDFs (FSDS, COC, field logbooks, digital photographs) to a dedicated FTP site.

4.0 FIELD DOCUMENTATION PROCEDURES

Field documentation serves as the primary foundation for all field data collected that will be used to evaluate the project site. There are two main forms of field documentation – field logbooks and FSDS forms. All field documentation shall be accurate, legible and written in indelible black or blue ink. Absolutely no pencils or erasures shall be used. Incorrect entries in the FSDS forms or field logbooks will be corrected by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction.

4.1 Field Logbooks

The field logbook shall be a bound, weatherproof book with numbered pages, and shall serve primarily as a daily log of the activities carried out during the fieldwork. All entries shall be made in indelible black or blue ink. A field logbook shall be completed for each operation undertaken during the field tasks. To further assist in the organization of the field log books, the project name and the date shall be recorded on top of each page along with the significant

activity description (e.g., surface sample or soil boring number). All original field documentation shall be retained in the project files.

Skipped pages or blank sections at the end of a field log book page shall be crossed out with an "X" covering the entire page or blank section; "No Further Entries," initials, and date shall be written by the person crossing out the blank section or page. The responsible field team member shall write his/her signature, date, and time after the day's last entry.

Field activities vary from project to project; however, the concept and general information that shall be recorded are similar. The descriptions of field data documentation given below serve as an outline; individual activities may vary in documentation requirements. A detailed description of two basic example logbooks, suitable for documentation of field activities, is given below. These field logbooks include the FTL logbook and the field geologist/sampling team logbook.

FTL Logbook: The FTL's responsibilities include the general supervision, support, assistance, and coordination of the various field activities. As a result, a large portion of the FTL's day is spent rotating between operations in a supervisory mode. Records of the FTL's activities, as well as a summary of the field team(s) activities, shall be maintained in a logbook. The FTL's logbook shall be used to fill out daily/weekly reports and daily quality control reports (DQCRs), and therefore, shall contain all required information. Entries shall be preceded with time in military units for each observation. Items to be documented include:

- Record of tailgate meetings
- Personnel and subcontractors on job site and time spent on the site
- Field operations and personnel assigned to these activities
- Site visitors
- Log of FTL's activities: time spent supervising each operation and summary of daily operations as provided by field team members
- Problems encountered and related corrective actions
- Deviations from the sampling plan and reasons for the deviations
- Records of communications; discussions of job-related activities with the client, subcontractor, field team members, and project manager

- Information on addresses and contacts
- Record of invoices signed and other billing information
- Field observations

Field Geologist/Sampling Team Logbook: The field geologist or sampling team leader shall be responsible for recording the following information in a logbook:

- Health and Safety Activities
  - Calibration records for health and safety equipment (e.g., type of PID, calibration gas used, associated readings, noise dosimeters, etc.)
  - Personnel contamination prevention and decontamination procedures
  - Record of daily tailgate safety meetings
- Weather
- Calibration of field equipment
- Equipment decontamination procedures
- Personnel and subcontractors on job site and time spent on the site
- Station identifier
- Sampling activities
  - Sample location (sketch)
  - Equipment used
  - Names of samplers
  - Date and time of sample collection
  - Sample interval
  - Number of samples collected
  - Analyses to be performed on collected samples
- Disposal of contaminated wastes (e.g., PPE, paper towels, Visqueen, etc.)
- Field observations
- Problems encountered and corrective action taken
- Deviations from the sampling plan and reason for the deviations
- Site visitors

4.2 Field Sample Documentation

Sample Labels: A unique sample identification label shall be affixed to all sample containers. All samples will be labeled in a clear, precise way for proper identification in the field and for tracking in the laboratory. At the time of collection, each sample will be labeled with a unique 5-digit sequential identification (ID) number, referred to as the Index ID. The Index ID for all samples collected as part of OU3 sampling activities will have a two-character prefix specific to the sampling Phase (e.g., Phase 1 samples will have a "P1" prefix, P1-12345) as specified in the applicable SAP. Index ID labels will be ½ inch x 1 ¾ inch in size and pre-printed for use in the field. For each Index ID, multiple labels will be printed to allow for multiple containers of the same sample (i.e., for different analyses).

Index ID Label Example:

P1-12345

Each collection container will be labeled with a container label that enables the field team member to record the container-specific details, such as the method of sample preparation (e.g., filtered/unfiltered), method of preservation, and the analytical methods that will be requested. Container labels will be 2 inch x 4 inch in size and pre-printed for use in the field. Any container-specific information shall be written in indelible ink.

Container Label Example:

Date/Time:_____

Index ID:

Media (circle one): AQ SO A BK DB TC MT
For AQ, Filtered? (circle one): Yes No
Container: _____
Preservation: _____
Analyses: _____
_____

Media acronyms: AQ – aqueous media, SO – solid media, A – air,
BK – tree bark, DB – organic debris, TC – tree age core, MT – mammal tissue

After labels have been affixed to the sample container, the labels will be covered with clear packaging tape to ensure permanence during shipping.

Any unused Index ID labels should be crossed out to avoid the possibility of using unused labels for a different sample.

Field Sample Data Sheet (FSDS) Forms: Data regarding each sample collected as part of the OU3 sampling will be documented using Libby-specific FSDS forms (provided as Attachment 1). These FSDS forms are medium-specific and designed to facilitate data entry of station location, sample details, and field measurements needed for the OU3 investigation.

In the field, one field team member will be responsible for recording all sample details onto the appropriate FSDS form. At the time of sample labeling, one Index ID label will be affixed to the FSDS form in the appropriate field. All written entries on the FSDS form shall be accurate, legible and written in indelible black or blue ink.

Once the FSDS form is complete, written entries will be checked by a second field team member. These two field team members will initial the bottom of the FSDS form in the appropriate field to document who performed the written data entry and who performed the QC check of the FSDS form.

On a weekly basis (or more frequently as conditions permit), information from the hard copy FSDS form will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate. The Field Sample/Data Manager and the FTL will initial in the appropriate field on the paper FSDS form to document who performed the data entry into the database and who performed the QC check.

4.3 Photologs

Photologs are often used in the field to document site conditions and sample location characteristics. While photographs may not always be required, they shall be used wherever

applicable to show existing site conditions at a particular time and stage of the investigation or related site activity. Photolog information shall include:

- station location identifier
- Index ID (if applicable)
- date and time of photo
- direction/orientation of the photo
- description of what the photo is intended to show

An engineer's scale or tape shall be included in any photographs where scale is necessary. Any wasted frames or images in a roll of film or sequence of digital images shall be so noted in the field logbook.

4.4 Chain-of-Custody Records

Custody Seals: Custody seals with the date and initials of the sampler will be used on each shipping container to ensure custody. The custody seal will be placed on opposites sides of the cooler across the seam of the lid and the cooler body. Alternatively, if the sample containers are all placed inside a liner bag within the cooler, the custody seal may be placed across the seal of the liner bag inside of the cooler.

Chain-of-Custody Forms: COC procedures allow for the tracking of possession and handling of individual samples from the time of field collection through to laboratory analysis. Documentation of custody is accomplished through a COC form that lists each sample and the individuals responsible for sample collection and shipment, sample preparation, and receipt by the analytical laboratory. The COC form also documents the analyses requested for each sample. Whenever a change of custody takes place, both parties will sign and date the COC form, with the relinquishing party retaining a copy of the form. The party that accepts custody will inspect the COC form and all accompanying documentation to ensure that the information is complete and accurate. Any discrepancies will be noted on the COC form. Shipping receipts shall be signed and filed as evidence of custody transfer between field sampler(s), courier, and laboratory.

Attachment 2 provides an example of the COC form that will be used for all samples collected as part of OU3 sampling. This form will be printed as a carbonless triplicate form to facilitate retention of COC copies by relinquishing parties. As seen, the COC form includes the following information:

- sample identifier (Index ID)
- date and time of collection
- method of sample preparation and preservation
- number of sample containers
- analyses requested
- shipping arrangements and airbill number, as applicable
- recipient laboratories
- signatures of parties relinquishing and receiving the sample

On a daily basis, the Field Sample/Data Manager will package samples for shipping, complete hard copy COC forms, and ship all samples as outlined in SOP No. 8. On a daily basis, information from the hard copy COC form necessary for sample tracking will be manually entered into a field-specific OU3 database using electronic data entry screens by the Field Sample/Data Manager. Once electronic data entry is complete, QC of all data entry will be completed by the FTL or their designate.

5.0 FIELD DATA TRANSMITTAL

Copies of all FSDS forms, COC forms, and field log books will be scanned and posted in portable document format (PDF) to a project-specific file transfer protocol (FTP) site daily. This FTP site will have controlled access (i.e., user name and password are required) to ensure data access is limited to appropriate project-related personnel. File names for scanned FSDS forms, COC forms, and field log books will include the sample date in the format YYYYMMDD to facilitate document organization (e.g., FSDS_20090831.pdf).

Electronic copies of all digital photographs will also be posted weekly (or more frequently as conditions permit) to the project-specific FTP site. File names for digital photographs will include the station identifier, the sample date, and photograph identifier (e.g., ST-1_20090831_12459.tif).

A copy of the field-specific OU3 database will be posted to the project-specific FTP site on a weekly basis (or more frequently as conditions permit). The field-specific OU3 database posted to the FTP site will include the post date in the file name (e.g., FieldOU3DB_20090831.mdb).

6.0 CORRECTIONS AND MODIFICATIONS

6.1 Field Deviations and Modifications

It is recognized that deviations and modifications from the standard operating procedures may be necessary based on site conditions. Any requested field modifications will be submitted by Robert Marriam (Remedium Group, Inc. - W.R. Grace contractor) to Bonita Lavelle (EPA Region 8 - Remedial Project Manager) for review and approval. All modification requests will be recorded in a Field Modification Approval Form (see Attachment 3).

6.2 Corrections to Hard Copy Forms

If an error is identified on an FSDS or COC form prior to entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. Data entry into the field-specific OU3 database and scanning/posting of the hard copy forms should proceed following the data entry procedures described above.

If an error is identified on an FSDS or COC form after entry into the field-specific OU3 database, the information should be corrected on the hard copy form by crossing out the incorrect entry with one line, the individual making the correction will initial and date next to the correction. The corrected form should be scanned and posted to the project-specific FTP site. File names for corrected FSDS forms will include the Index ID of the corrected sample to facilitate document organization (e.g., FSDS_C_P1-12345.pdf). File names for corrected COC

forms will include the COC ID of the corrected COC form to facilitate document organization (e.g., COC_C_OU3-36512.pdf). Necessary data corrections will be made to the master OU3 database by the database manager.

If changes are made to a COC form, the analytical laboratory should be provided with a corrected COC form.

7.0 REFERENCES

Agency for Toxic Substances and Disease Registry. 2006. Asbestos Exposure and Your Health.

RCRA Ground-Water Monitoring: Draft Technical Guidance, November 1992.

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ATTACHMENT 1

OU3 FIELD SAMPLE DATA SHEET (FSDS) FORMS

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Sheet No.: AA2-_____

LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2
STATIONARY AMBIENT AIR MONITOR

☐ Check box if GPS information has been recorded previously

Field Logbook No: _____ Page No: _____

Station ID: _____ Station Comments: _____

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: _____ Y coord: _____ Elevation: _____m

Sampling Team: _____ Sampler Initials: _____

Data Item	Cassette 1	Cassette 2	Cassette 3
Index ID	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*
Sample Height (ft)			
Location Description			
Field QC Type (circle)	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID: _____	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID: _____	FS-(field sample) FB-(field blank) FD-(field dup) For FD, Parent ID: _____
Matrix Type	Outdoor	Outdoor	Outdoor
Flow Meter Type	Rotameter	Rotameter	Rotameter
Archive blank (circle)	Yes No	Yes No	Yes No
Pump ID Number			
Flow Meter ID Number			
Start Date (mm/dd/yy)			
Start Time (hh:mm)			
Start Counter			
Daily Flow Check: Record time (hh:mm) and flow rate (L/min) in fields provided	Check1 / Time / Flow Check2 Check3 Check4	Check1 / Time / Flow Check2 Check3 Check4	Check1 / Time / Flow Check2 Check3 Check4
Stop Date (mm/dd/yy)			
Stop Time (hh:mm)			
Stop Counter			
Pump fault? (circle)	Yes No	Yes No	Yes No
Stop Flow (L/min)			
Field Comments Cassette Lot Number: _____			
Entered By (Provide initials):		**Validated By (Provide initials):**	

For Data Entry Completion (Provide Initials)	Completed by:	QC by:

Sheet No.: FSB1-_____

LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2
FOREST SOIL AND TREE BARK

Field Logbook No: _____ Page No: _____

Station ID: _____ Sampling Date: _____

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: _____ Y coord: _____ Elevation: _____m

Sampling Team: _____ Sampler Initials: _____

Station Comments: _____

TREE BARK SAMPLES

Index ID:	**Field QC Type (circle one):** FS (field sample) FD (field duplicate) For FD, Parent ID: _____	**Sample Area (cm^2):** _____	**Tree Species:** _____ **Collection Height (ft):** _____ **Diameter* (in):** _____	**Age Core Collected? (circle one):** Y N
Index ID:	**Field QC Type (circle one):** FS (field sample) FD (field duplicate) For FD, Parent ID: _____	**Sample Area (cm^2):** _____		
Field Comments:				
Entered by (Provide initials):		**Validated by (Provide initials):**		

*Measured with "D-tape"

FOREST SOIL SAMPLES

Index ID:	**Field QC Type (circle one):** FS (field sample) FD (field duplicate) For FD, Parent ID: _____	**Bulk Soil Description** **Depth (in)** **Start:** _____ **End:** _____	**Bulk Soil Description** **Sample Type:** Grab Composite **# of Comp.:** ___	**Organic Debris Collected? (circle one):** Y N
Index ID:	**Field QC Type (circle one):** FS (field sample) FD (field duplicate) For FD, Parent ID: _____	**Bulk Soil Description** **Depth (in)** **Start:** _____ **End:** _____	**Bulk Soil Description** **Sample Type:** Grab Composite **# of Comp.:** ___	**Organic Debris Collected? (circle one):** Y N
Index ID:	**Field QC Type (circle one):** FS (field sample) FD (field duplicate) For FD, Parent ID: _____	**Bulk Soil Description** **Depth (in)** **Start:** _____ **End:** _____	**Bulk Soil Description** **Sample Type:** Grab Composite **# of Comp.:** ___	**Organic Debris Collected? (circle one):** Y N
Field Comments:				
Entered by (Provide initials):		**Validated by (Provide initials):**		

For Data Entry Completion (Provide Initials)	Completed by	QC by

Sheet No.: GW2- _____

LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev3

GROUNDWATER

☐ Check box if GPS information has been recorded previously

Field Logbook No: _____ Page No: _____

Station ID: _____ Station Alias: _____ Sampling Date: _____

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

X coord: _____ Y coord: _____ Elevation (meters): _____

Sampling Team: _____ Sampler Initials: _____

Station Comments: _____

Well: Measuring Point (MP) of Well: _____ MP Units: _____

Screened Interval (ft BGL): _____ Filter Pack Interval (ft BGL): _____

Casing Stickup (ft) _____ Sample Intake Depth (ft BMP): _____

Calibration:

☐ Daily Verification Weekly Date: _____

VOA Vial pH: _____ Other pH: _____

Purge Method:

☐ Dedicated Submersible (SP) ☐ Portable Bladder (BP) ☐ Peristalic (PP)

☐ Portable Submersible (SP) ☐ Dedicated Bailer (B) ☐ Grab (G)

☐ Dedicated Bladder (SP) ☐ Disposable Bailer (B) ☐ Other: _____

Multiplication Factors:

1" = 0.04 2" = 0.16

3" = 0.37 4" = 0.65

6" = 1.47

Purge:

Starting Water Level (ft BMP): _____ Total Depth (ft BGL): _____ Water Column Height (ft): _____

Casing Diameter (in ID): _____ Multiplication Factor: _____ Casing Volume (gal): _____ 2X: _____ 3X: _____ 4X: _____

Water Level (ft BMP) at End of Purge: _____ Total Depth (ft BMP) at End of Purge: _____

Time (hh:mm)	Temp. (°C)	pH	Spec. Cond. (mS/cm@25°C)	ORP (mV)	Diss. O_2 (mg/L)	Turbidity (NTU)	Vol. Evac. (gal)	Flow Rate (gal/min)	Comments
Final Parameters									

Data Item	**Sample 1**	**Sample 2**	**Sample 3**
Index ID	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*
Field QC Type (circle one):	FS SP FD MS MSD PE EB FB TB Parent ID: _____	FS SP FD MS MSD PE EB FB TB Parent ID: _____	FS SP FD MS MSD PE EB FB TB Parent ID: _____
Field Comments:			
Cooler:			
Entered by (Provide initials):		**Validated by (Provide initials):**	

Note:

FS	Field Sample	**SP**	Field Split Sample	**FD**	Field Duplicate Sample
TB	Trip Blank Sample	**MS**	Matrix Spike Sample	**MSD**	Matrix Spike Duplicate Sample
FB	Field Blank Sample	**EB**	Equipment Decon Blank Sample	**PE**	Performance Evaluation Sample

For Data Entry Completion (Provide Initials)	Completed by	QC by

LIBBY OU3 FIELD SAMPLE DATA SHEET
ACTIVITY-BASED SAMPLING (ABS) PERSONAL AIR MONITOR

ABS Area: ABS-_____ **Sampling Date: _____** **Sampling Team:** MWH

Person #1 Name: _____ **Index ID:** *AFFIX LABEL HERE*

Person #2 Name: _____ **Index ID:** *AFFIX LABEL HERE*

Field Blank Index ID: *AFFIX LABEL HERE* **Cassette Lot Number: _____**

Field Logbook Number: _____ **Field Logbook Pages: _____**

ABS Activity	Activity Sample Time (hh:mm)		Rotometer Flow (L/min)			
			Person #1		Person #2	
	Start	Stop	Start	Stop	Start	Stop
ATV Riding						
Hiking						
Sawing/Stacking						
Raking/Digging						
Fire						

Person #1 Pump ID No.: _____ Rotometer ID No.: _____ GPS ID No.: _____
Person #2 Pump ID No.: _____ Rotometer ID No.: _____ GPS ID No.: _____

Field Comments:

Weather Description--

Other--

Field Data Entered by:		Field Entries Checked by:	

Database Entry:		*Database QC:*

Sheet No.: SM-_____

LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS) rev2
SOIL-LIKE MATERIALS

Field Logbook No: _____ Page No: _____

Station ID: _____ Sampling Date: _____

GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters

Sampling Team: _____ Sampler Initials: _____

Station Comments: _____

Data Item	Sample 1	Sample 2	Sample 3
Index ID	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*	*AFFIX LABEL HERE*
Matrix (circle one):	Surface Soil Tailings Waste Rock Roadway Other _____	Surface Soil Tailings Waste Rock Roadway Other _____	Surface Soil Tailings Waste Rock Roadway Other _____
Sample Time (hh:mm)			
Sample Type (circle one):	Grab Composite # of Comp: _____	Grab Composite # of Comp: _____	Grab Composite # of Comp: _____
Sample Depth	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____	Start Depth (in): _____ End Depth (in): _____
Field QC Type (circle one):	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____	FS (field sample) FD (field duplicate) For FD, Parent ID: _____ TB (trip blank) Cooler: _____ PE (perf. eval.) ID: _____
Transect Start Location or Grab Sample Location	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m
Transect End Location	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m	X coord: _____ m Y coord: _____ m Elevation: _____ m
Field Comments:			
Cooler:			
Entered by (Provide Initials):		**Validated by (Provide Initials):**	

For Data Entry Completion (Provide Initials)	Completed by	QC by

Sheet No.: SWS- ________
SED- ________

LIBBY OU3 FIELD SAMPLE DATA SHEET
SURFACE WATER AND SEDIMENT

Station ID: ________ **Sampling Date:** ________
Field Logbook ID: ________ **Logbook Page No:** ________
GPS Coordinate System: UTM Zone 11 North, NAD83 datum, meters
For New Stations Only: **X coord:** ________ **Y coord:** ________ **Elev:** ________
Sampling Team: MWH **Samplers Initials:** ________

WATER QUALITY PARAMETERS (if applicable)

Time Measured (hh:mm)	Temp. (°C)	pH	Specific Conductance (mS/cm Auto-comp @ 25°C)	Diss. O_2 (mg/L)	ORP (mV)	Turbidity (NTU)

SAMPLE COLLECTION

Index ID	*AFFIX LABEL HERE*	**Sampling Time:** ________ **Sample Type: Field Sample** **Media : Surface Water Sediment**	**Sampling Method** (if applicable): **Grab or Composite** **# of Composites:** ________ **Sampling Depth: Top (in)** ________ **Bot (in)** ________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:** ________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:** ________ **Sampling Depth: Top (in)** ________ **Bot (in)** ________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:** ________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:** ________ **Sampling Depth: Top (in)** ________ **Bot (in)** ________
Index ID	*AFFIX LABEL HERE*	**Sampling Time:** ________ **Sample Type: SP FD** **MS MSD PE** **FB TB EB** **Media : Surface Water Sediment**	**Sampling Method (if applicable):** **Grab or Composite** **# of Composites:** ________ **Sampling Depth: Top (in)** ________ **Bot (in)** ________

COMMENTS

Note:

FS	Field Sample	**SP**	Field Split Sample	**FD**	Field Duplicate Sample
TB	Trip Blank Sample	**MS**	Matrix Spike Sample	**MSD**	Matrix Spike Duplicate Sample
FB	Field Blank Sample	**EB**	Equipment Decon Blank Sample	**PE**	Performance Evaluation Sample

Field Data Entered by:		**Field Entries Checked by:**	

Database Entry:	*Database QC:*

FSDS Rev. 0

Sheet No.: SM- _____

LIBBY OU3 FIELD SAMPLE DATA SHEET (FSDS)
SMALL MAMMAL TISSUE COLLECTION

Field Logbook ID: _____ **Logbook Page No**: _____

Necropsy Date: _____ **Personnel Initials**: _____

Small Mammal Field ID: **SM-**_____-_____-_____-_____ **Animal Weight (grams):** _____(w/o fetuses if pregnant)

*[**SM** - station ID - transect ID - trap# - animal#]*

General Necropsy Comments: _____

	TISSUE #1	TISSUE #2	TISSUE #3	TISSUE #4
Tissue Type (circle one):	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____
Weight (mg):				
Index ID:	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*
Field QC Type (circle one):	FS FD TB	FS FD TB	FS FD TB	FS FD TB
Tissue Comments:				

	TISSUE #5	TISSUE #6	TISSUE #7	TISSUE #8
Tissue Type (circle one):	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____
Weight (mg):				
Index ID:	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*
Field QC Type (circle one):	FS FD TB	FS FD TB	FS FD TB	FS FD TB
Tissue Comments:				

	TISSUE #9	TISSUE #10	TISSUE #11	TISSUE #12
Tissue Type (circle one):	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____	TY AR ES ST SIN LIN LU EY CAR Other:_____
Weight (mg):				
Index ID:	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*	*Affix Label Here*
Field QC Type (circle one):	FS FD TB	FS FD TB	FS FD TB	FS FD TB
Tissue Comments:				

Tissue Type Descriptors: TY = thyroid; ARD = adrenal gland; ES = esophagus; SIN = small intestine; LIN = large intestine; LU = lung; EY = eyeball; CAR = carcass

Field QC Type Descriptors: FS = Field Sample; FD = Field Duplicate; TB = Tissue Blank

For Data Entry Completion (Provide Initials)	Completed by	QC by

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ATTACHMENT 2

OU3 CHAIN OF CUSTODY FORM

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LIBBY OU3 – CHAIN-OF-CUSTODY RECORD/REQUEST FOR ANALYSIS

COC No. ______________

PAGE: ______ OF: ______

ENTERED BY (Signature): ____________________ PROJECT MANAGER: ____________________ DATE: ______________

METHOD OF SHIPMENT: ____________________ CARRIER/WAYBILL NO.: ______________ DESTINATION: ____________________

SAMPLES							ANALYSIS REQUEST																													
							Asbestos			Non-Asbestos (a)																										
Index ID	Date	Time	Media*	Air Volume (L) or Tree Bark Sample Area (cm^2)	Filtered	Archive	TEM-ISO 10312 (b,c)	PLM (d)		TAL Metals+Boron	Mercury	TOC	DOC	Paste pH	Fluoride	Chloride, Sulfate	Total Phosphorus	Cyanide	VPH	EPH	OPP Pesticides	Chlorinated Pesticides	Herbicides	PCBs	SVOCs	VOCs	TDS, TSS, Nitrite, Alkalinity	Ammonia, Nitrate, TKN	Orthophosphate	Radiochemistry	Radium, Uranium	Hardness			Age core (e)	Remarks

	TOTAL NUMBER OF CONTAINERS	LABORATORY COMMENTS/CONDITION OF SAMPLES	Cooler Temp:

RELINQUISHED BY:			DATE	TIME	RECEIVED BY:		
SIGNATURE	PRINTED NAME	COMPANY			SIGNATURE	PRINTED NAME	COMPANY

** Media: AQ - Aqueous SO – Solid A –Air BK – Tree Bark DB – Organic Debris (Duff) TC – Tree Age Core MT – Mammal Tissue*

Notes --

(a) Method, container, and preservation details are provided in the attached tables

(b) With Libby-specific modifications. See applicable O3 SAP for counting and stopping rules

(c) See applicable SAP for details on preparation methods.

(d) Preparation by ISSI-LIBBY-01 and analysis by SRC-LIBBY-01 (PLM-Grav) and SRC-LIBBY-03 (PLM-VE)

(e) In accordance with procedures in Phipps (1985).

DISTRIBUTION: PINK: Field Copy YELLOW: Laboratory Copy WHITE: Return to Originator

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ATTACHMENT 3

OU3 FIELD MODIFICATION APPROVAL FORM

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FIELD MODIFICATION APPROVAL FORM
LFM-OU3-_____
Libby OU3 Phase ___ Sampling & Analysis Plan

Requested by:_____ Date:_____

Description of Deviation:

☐ EPA Region 8 has reviewed this field modification approves as proposed.

☐ EPA Region 8 has reviewed this field modification and approves with the following exceptions:

☐ EPA Region 8 has reviewed this field modification and does not agree with the proposed approach for the following reasons:

_____ _____

Bonita Lavelle, EPA RPM Date

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ATTACHMENT C

Product Literature and Specifications for Geotech, Inc.
GeoPump® Series II Peristaltic Pump

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Click to go to www.geotechenv.com

geotech

Peristaltic Sampling Pumps

Geopump™ Peristaltic Pumps

The Geotech Series I and II Geopump™ Peristaltic Pumps are designed for single and multi-stage pressure or vacuum pumping of liquids. The Geopump is ideally suited for field sample removal from shallow wells and all surface water sources or laboratory use.

FEATURES

- **Exceptional field durability**
- **Operate from 60 to a maximum of 600 RPM**
- **Delivery rate of 1.67 ml per revolution**
- **Operate to a depth of 27 feet at sea level**
- **Variable speed control**
- **Reversible flow feature for back-flushing**
- **Disposable and dedicated tubing means controlled costs and no decontamination issues**

Geopump™ Peristaltic Pump Series II with EZ-load 2 pump head (optional), 5 ft tubing, carrying case and power cord

OPERATION

The Geotech Peristaltic Pumps operate by mechanical peristalsis, so the sample only comes in contact with the tubing. This allows for sample integrity as well as easy cleaning and replacement. With the optional stainless steel tubing weight, tubing can be lowered to a specific depth without curling or floating on the surface of the water. Geopumps operate from any external 12 VDC or 120 VAC power source.

SERIES I Geopump™ Peristaltic Pumps are available in AC only, DC only, or an AC/DC combination. These units have one pumping station which can be piggy-backed for multi-station pumping. They have variable speeds ranging from 60 RPM to 350 RPM.

SERIES II Geopump™ Peristaltic Pumps are available in AC only, DC only, or an AC/DC combination. They have two pumping stations which can also be piggy-backed for multi-station pumping. The first pumping station has a variable speed of 30 to 300 RPM and the second station 60 to 600 RPM.

Geopump™ Peristaltic Pump Series I with EZ-load 2 pump head (optional) and dispos-a-filter capsule

Geotech Environmental Equipment, Inc.
2650 East 40th Avenue • Denver, Colorado 80205
(303) 320-4764 • **(800) 833-7958** • FAX (303) 322-7242
email: sales@geotechenv.com website: www.geotechenv.com

geotech

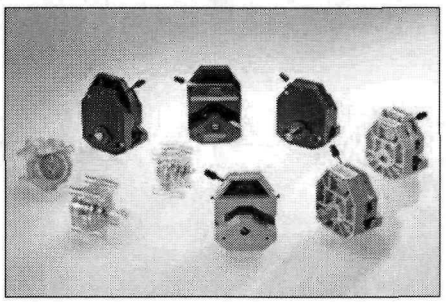
Peristaltic Sampling Pumps

Geopump™ Peristaltic Pump Specifications

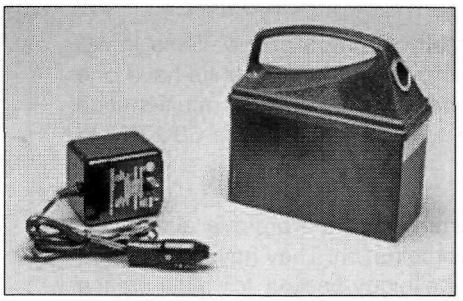
Operating range	Suction from 27 feet at sea level
Principle of operation	Mechanical peristalsis
Dimensions	3.5 x 8 x 8 inches
Power source	Any external 12 V DC or 120 V AC
Power cord	12 V DC adapter cord or standard AC power cord
Power cord length	AC cord: 8 feet; DC cord: 15 feet
Range of speed: Series I	60 to 350 rpm
Range of speed: Series II	First pumping station 30 to 300 rpm second pumping station 60 to 600 rpm
Speed control	Stepless variable speed control
Liquid delivery rate	1.67 ml per revolution
Pumping options	Pressure or vacuum (reversible flow)
Pump head rotor	Cold rolled steel
Warranty	1 year
Basic system kit	Geopump (as specified), 5ft of tubing, power cord (as specified), field case, and manual. Pump head sold separately: standard, easy-load 1, easy-load 2, or quick load.

Accessories

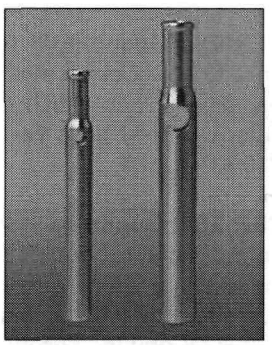
- Quick Load pump head
- Easy Load pump head
- Silicone tubing
- Tygon tubing
- Other tubing
- Stainless steel tubing weight
- Custom length power cord
- Geotech Back Flushing Membrane Filter Holder
- Geotech In-line Dispos-a-filter
- Rechargeable battery
- Battery charger
- Optional stainless steel rotor for pump
- Additional power cords
- Carrying case



Geopump™ pump heads (easy-load, standard, quick load) shown with small and large shaft



Geopump™ Modular Battery and Charger



Geopump™ Tubing Weights

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email: sales@geotechenv.com website: www.geotechenv.com

ATTACHMENT C

Sediment Sample Preparation and Analysis Standard Operating Procedures
Lab Modification

ISSI-LIBBY-01 (Rev. 10)
SRC-LIBBY-01 (Rev. 2)
SRC-LIBBY-03 (Rev. 2)
Libby OU3 Water PCM Analysis Mod 1
LB-000029b

NOTE: This SOP has been prepared for use at the Libby Asbestos Superfund Site. The applicability of this SOP at other sites should be evaluated by the site team with regard to site-specific goals and objectives.

Date: December 6, 2007 SOP No. ISSI-LIBBY-01 (Rev. 10)

Title: SOIL SAMPLE PREPARATION

SYNOPSIS: A standardized method for preparation of soil samples for asbestos analysis at the Libby Asbestos Superfund Site is described.

Original Author: William Brattin Syracuse Research Corporation[1]

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8:		12/11/07
Syracuse Research Corp.	WS Brattin	12/10/07

[1] This SOP was originally prepared by ISSI Consulting Group. ISSI is no longer in existence, and finalization of the SOP was performed by Syracuse Research Corporation (SRC).

REVISION LOG

Revision Number	Revision Date	Reason for Revision
1	1/7/00	Incorporation of sieving to the sample preparation.
2	7/12/00	Revision in sieve size, other minor edits.
3	5/7/02	Incorporate minor edits
4	8/1/02	Modify sieving procedure, add grinding step
5	3/6/03	Incorporate modifications to the procedure and documentation requirements
6	3/24/03	Incorporate modifications to the log-sheets to conform with electronic data storage requirements and add grinder blank requirements.
7	8/5/03	Incorporate modifications to drying and sample storage procedures
8	5/4/04	Incorporate modifications to drying batch size and recording of preparation information
9	5/14/07	Incorporate modifications so as to expand use to other Operable Units (removed references to OU4 / CSF, changed Index ID to Sample ID). Repair formatting. Remove reference to missing Figure 1. Add optional use of electronic logs. Oven temperature set to 90±10 degrees C. Lowered inventory batch size from ~120 to ~50 samples so that one inventory batch can fit in one tub. Designate drying batch as one batch per oven (~20 samples). Allow for optional use of disposable drying pans. Remove direction to NOT move grinding plates during decontamination (new BICO design allows plates to be separated for decontamination without adjusting gap). Ovens will be calibrated daily. *[Note: Revision 9 was an unsigned version that reflects changes made at the Troy Preparation Laboratory. Some of the changes in Revision 9 are retained in Revision 10, below].*
10	12/06/07	Incorporate modifications so as to expand use to other Operable Units. Designate drying batch as ~20 samples. Allow for optional use of disposable drying pans. Allow alternative methods for decontamination of plate grinder. Clarify and modify QC requirements. General editing for clarity.

1.0 PURPOSE

This Standard Operating Procedure (SOP) has been prepared by the United States Environmental Protection Agency (USEPA) Region 8 to standardize the methods used to prepare soil samples from the Libby Asbestos Superfund Site for the analysis of asbestos content. This procedure is intended for use by employees of USEPA Region 8 and by contractors and subcontractors supporting USEPA Region 8 projects and tasks for the Remedial Investigation work performed at the Libby site. Deviations from the procedures outlined in this document must be reviewed and approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist.

2.0 RESPONSIBILITIES

Each laboratory that performs soil preparation activities under this SOP must have a designated Preparation Laboratory Project Leader (PL^2). The PL^2 may be an USEPA employee or contractor. The PL^2 is responsible for ensuring that all personnel in the laboratory who perform work under this SOP are familiar with the SOP, and for ensuring that all work performed satisfies the requirements of this SOP and any other relevant laboratory-specific operating procedures. It is also the responsibility of the PL^2 to communicate and document the need for any deviations from the SOP with the appropriate USEPA Region 8 Remedial Project Manager or Regional Chemist.

All laboratory personnel preparing Libby soil samples are responsible for reading and understanding the requirement of this SOP, and for performing all applicable tasks in accordance with this SOP. Any laboratory worker who identifies any issues or encounters any difficulties in implementation of this SOP is responsible for promptly communicating the issue or difficulty to the PL^2. In addition, all laboratory personnel are responsible for reading and understanding the Health and Safety Plan (HASP) applicable to the soil preparation activities in that laboratory, and performing all tasks in accord with the requirements of that HASP.

3.0 EQUIPMENT

- General purpose laboratory oven - capable of maintaining a constant temperature of approximately 90°C.
- Analytical balance - capable of measuring in a range of 0.1 g to at least 2000 g, calibrated and accurate to the tolerance limits indicated in Attachment 2.
- Riffle splitter - with 3/4 inch chutes to split samples.

- Plate grinder - capable of accepting soil particles of approximately 1/4 inch diameter and grinding to produce particles of approximately 250 µm.

- HEPA Vacuum - A portable vacuum unit equipped with a high efficiency particulate air (HEPA) filter to remove any asbestos fibers and other soil particles from the exhaust air. Used to decontaminate equipment and maintain general laboratory cleanliness.

- Metal scoop or spoon - for transferring samples. Plastic scoops or spoons are not acceptable.

- 1/4 inch metal sieve and catch pan - for coarse sieving samples. Plastic sieves and pans are not acceptable.

- 60 mesh (250 µm) and 200 mesh (74 µm) metal sieves - for verification of the plate grinder settings. Plastic sieves are not acceptable.

- Clean quartz sand - required for preparation of grinding and drying blank samples and for decontamination of grinder.

- Clean soil - required for calibration of grinder.

- Drying pans with lids - used during the sample drying process, lids used to cover samples during transfer

- Sample containers - plastic ziplock bags (pint and gallon size).

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Personal Protective Equipment - as specified in the applicable Health and Safety Plan for the soil preparation laboratory.

- Laboratory notebook and pen - used to record progress, any problems or observations and deviations. All information in the laboratory notebook must be recorded in pen (not pencil).

- Sample Drying Log Sheets - (Attachment 1). Used to record all sample drying information.

- Sample Preparation Log Sheets - (Attachment 1). Used to record all sample preparation information (splitting, sieving and grinding).

- Equipment Calibration and Maintenance Logs for:

  - Analytical Balance (Attachment 2)
  - Plate Grinder (Attachment 3)
  - Ventilation Hood (Attachment 4)
  - HEPA Vacuum (Attachment 5)
  - Drying Oven (Attachment 6)

  These logs are used to record all maintenance and calibration records for the listed equipment. If hard copy, all entries must be recorded in pen, and the logs must be organized and maintained in a laboratory notebook.

- Sample Labels – Self-adhesive labels for attachment to sample bags.

- Trash Bags - used to dispose of gloves, wipes and other investigation derived waste.

- Indelible Marking Pen - used to record sample information onto plastic ziplock bags and to record logbook information.

4.0 METHOD SUMMARY

Figure 1 provides an overview of the steps in the soil preparation process. Soil samples received from the field are first dried in a laboratory oven and are then split into a preparation sample and an archive sample. The preparation sample is sieved to separate coarse material (> 1/4 inch) from fine material (< 1/4 inch). The fine material is ground to a particle size of less than 250 µm, and this fine ground material is split into several aliquots. This grinding step is needed to achieve a reasonable degree of homogeneity in the sample, and to allow for preparation of slides for microscopic analysis. The coarse fraction (if any) and one aliquot of the fine ground material are then sent to an analytical laboratory for asbestos analysis by methods specified in the project-specific Sampling and Analysis Plan. At present, the fine-ground sample is generally analyzed by Phase Contrast Microscopy (Visual Area Estimation) (PLM-VE) in accord with the most recent version of SOP SRC-LIBBY-03, and the coarse material is examined by stereomicroscopy and any observable particles of asbestos are removed and weighted in accord with the most recent version of SOP SRC-LIBBY-01.

It should be noted that this preparation method, coupled with these analytical techniques, is intended to estimate the total mass fraction of asbestos that is present in a sample, without regard

to the current size distribution of the asbestos particles. That is, no distinction is drawn between asbestos that is presently in a large "lump" that is non-respirable and free asbestos fibers that are readily released to air and inhaled. Because of this, concentration values based on this approach may tend to overestimate the amount of currently releasable fibers, but do provide an estimate of the total amount of fibers that may be releasable in the future.

5.0 SOIL STORAGE

Upon receipt at the soil preparation facility, samples will be grouped into an inventory batch of 50-120 samples. Samples will be archived according to the inventory batch they are assigned to and filed by the Inventory Batch ID (box number) noted in the Sample Drying Log and Sample Preparation Log (Attachment 1).

6.0 BULK SOIL DRYING

6.1 Equipment Calibration

Samples will be weighed prior to and following drying activities. The analytical balance used for drying activities will be calibrated on days when samples are loaded into, or unloaded from, the oven. Before weighing samples, calibrate the balance using S-1 class weights and record all measurements, any required maintenance, and the balance number in the Analytical Balance Calibration and Maintenance Log (Attachment 2).

All drying activities will be performed under a negative pressure HEPA filtered hood or similar containment box. Prior to loading the oven, the ventilation hood will be calibrated to ensure that the ventilation system is operating properly. Ventilation hood calibration and any required maintenance will be documented in the Ventilation Hood Calibration and Maintenance Log (Attachment 4).

A HEPA vacuum will be used to decontaminate the oven following the removal of dried samples. Vacuum calibration will be performed daily, prior to drying activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance Log (Attachment 5).

Oven temperature calibration will be performed on a daily basis (during periods of operation). Oven temperature calibration and any required maintenance will be documented in the Oven Temperature Calibration and Maintenance Log (Attachment 6).

6.2 Drying Procedure

- Prior to unsealing and drying each sample, record on the Sample Drying Log the starting sample mass to the nearest 0.1 g. Include the technicians initials and the date.

- Group samples into drying batches of approximately 20 samples per batch. Assign each batch a drying batch number, and record this number on the Sample Drying Log, along with the SOP and Revision Number and the oven number used to dry the samples.

- Include one preparation blank in each drying batch. See Section 12.1 for more details regarding preparation blanks.

- Set the oven temperature to approximately 90±1°C. For every drying batch, check the oven temperature to verify that proper temperature[2] has been reached and document the start date/time and temperature in the Sample Drying Log.

- Transfer each sample to be dried from its ziplock storage bag into a clean drying pan. Each sample should be transferred to its respective drying pan under the negative pressure HEPA filtered hood. Label each drying pan with the Index ID[3] of the sample. Place each sample in the oven.

- Leave the samples in the oven for approximately 24-48 hours or until completely dry. Verify that each sample is dry by squeezing a portion of the soil with a freshly gloved thumb and forefinger to test the cohesiveness. Once it is confirmed that samples are dry, record the technician's initials, and the date and time of completion, in the Sample Drying Log.

- Turn off the oven and allow the samples to cool in the oven. Once the samples are cooled, unload each sample and transfer each sample volume to a clean ziplock bag, re-bag the sample with another clean ziplock bag and identify the dried sample with the Index ID. All samples should be transferred to ziplock bags under the negative pressure HEPA filtered hood to prevent potential exposure to fibers that might be released from the sample.

- Record the sample mass of each dried and bagged sample to the nearest 0.1 g along with the technician's initials and the date in the Sample Drying Log.

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[2] Drying temperatures in the range of 80-100°C will not compromise sample integrity, but monitoring of oven temperature to ± 1°C is needed to allow early detection of any problems with the oven temperature control.

[3] Unique sample identifiers at the Libby site are referred to as "Index ID" numbers rather than "Sample ID" numbers. However, the meaning is the same.

6.3 Decontamination

Decontaminate the inside of the hood and the inside of the drying oven by HEPA vacuuming and wet wiping all surfaces before loading a new batch for drying.

If drying pans are to be re-used, decontaminate all sample drying pans under the ventilation hood using compressed air and a HEPA vacuum to remove any residual organic material left on the pans. Wet wipe or brush off any visible material that is not removed using the vacuum.

7.0 DIVISION OF ARCHIVE AND PREPARATION SAMPLES

All dried samples are mixed and split into two portions: one portion is held in archive, and the second portion is prepared for asbestos analysis. The sections below describe the sample splitting procedure.

7.1 Equipment Calibration

Prior to any splitting, sieving, or grinding activities, calibrate the ventilation hood to ensure that the ventilation system is operating properly. Document ventilation hood calibration and any required maintenance in the Ventilation Hood Calibration and Maintenance Log.

7.2 Procedure for Sample Splitting

Splitting must be performed in the hood to prevent potential exposure to fibers that might be released from the sample. Samples will be divided using the following steps:

- Place the cooled, re-bagged samples in the hood, and knead the contents of the bag to break up any soil clumps.
- Place one collection pan on each side of the riffle splitter. Pour the sample from its plastic bag through the splitter in order to divide the sample into two equal sub-parts.
- After splitting, set aside one portion for sample preparation, as described below. If the mass of the portion for preparation is larger than about 200 grams, split the preparation sample again so that 3/4 of the original sample will be archived and 1/4 will be set aside for processing.
- Place the remaining portion(s) into a clean, ziplock bag, re-bag the sample in another clean ziplock bag, and store as an archive sample in the event additional analyses are required in the future. Identify the archive sample with the Index ID and the suffix "A" (for archive fraction). Record the technician's initials and date in the Sample Preparation

Log. Store the archive portion in the numbered inventory box noted in the Sample Preparation Log.

7.3 Preparation Duplicate Samples

One preparation duplicate sample will be prepared for every 20 field samples processed. A preparation duplicate is generated by using the riffle splitter to divide the preparation fraction into two equivalent portions ("parent" and "duplicate"). The duplicate portion is assigned an independent Index ID and both the parent sample and the duplicate sample are then processed in an identical fashion and are each submitted to the laboratory blind. For further information on preparation and processing of preparation duplicates, refer to Section 12.4.

7.4 Performance Evaluation Samples

Performance Evaluation (PE) samples are used to assess the accuracy of the analytical laboratory and to check for any potential contamination or loss of asbestos during processing. For further information on preparation and processing of PE samples, refer to Section 12.3.

7.5 Decontamination

The splitter need not be decontaminated following this step if the next use of the splitter will be the division of the fine ground fraction of the same samples into four fractions (see Section 10, below). If for any reason the next use of the splitter is division of material from a different sample, the riffle splitter must be decontaminated as follows.

- Use a HEPA vacuum and compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the air blast. The splitter is now ready to process the next sample.

8.0 SIEVING THE PREPARATION SAMPLE

All preparation samples are sieved prior to grinding to separate out the coarse and fine fractions. The sample sieving procedure is described in the sections below.

8.1 Equipment Calibration

All sieving activities will take place in the hood. Refer to Section 6.1 for details regarding the frequency of ventilation hood calibration.

Samples are weighed during sieving activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance,

and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

8.2 Sample Sieving Procedure

Samples will be sieved using the procedure outlined below.

- Pour the sample onto a clean 1/4 inch stainless-steel sieve with a clean pre-weighed catch pan. Shake the screen until all particles <1/4 inch in size have passed through the screen into the pan. When needed, a pestle may be used to gently break up any remaining soil clumps to ensure all particles <1/4 in size pass through the screen.

- Pour all material which does not pass through the screen (>1/4 inch) into a new, tared, sample bag. This is the Coarse Fraction.

- Weigh and record the mass of the coarse fraction to the nearest 0.1 g in the Sample Preparation Log and record the technician's initials and the date. If all of the material passes through the screen, such that there is no coarse fraction, record a mass of zero for the coarse fraction in the Sample Preparation Log.

- Double-bag the coarse sample portion and identify the sample with the Index ID and "C" suffix on the sample bag. Coarse fraction samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed.

- All material that passes through the 1/4 inch screen is the Fine Fraction. Weigh and record the mass of the fine fraction to the nearest 0.1 g in the Sample Preparation Log.

Whenever possible, immediately process the fine fraction material in accord with the approach described in Section 9.3 (below). If processing cannot occur immediately, pour the fine fraction material into a new ziplock bag and identify the fine sample material with the Index ID and the suffix "F" (for "fine fraction"). Double-bag the sample and identify the sample with the Index ID and suffix on the outside of the bag.

8.3 Decontamination

All non-disposable pans and sieves will be decontaminated between samples. Decontaminate sieves and pans (and the pestle, if used) under the ventilation hood using compressed air. Wipe or brush off any visible material that is not removed from the air blast. A HEPA vacuum may also be used to remove any residual material.

9.0 GRINDING THE FINE FRACTION

The fine fraction of each preparation sample will be ground to produce a material of about 250 µm[4]. The procedure for grinding the fine fraction is outlined below.

9.1 Equipment Calibration

All grinding activities will take place in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

A HEPA vacuum will be used to decontaminate the hood and processing equipment, following the preparation of each sample. Vacuum calibration will be performed daily, prior to grinding activities. All system checks, required maintenance and the vacuum number will be recorded in the Vacuum Maintenance.

A plate grinder will be used to process samples. The grinder will be calibrated daily or after any adjustments are made to the plates. To verify proper particle size (approximately 250 µm), and demonstrate that samples will not be over-processed, grind a sample of clean soil (rather than quartz sand) and sieve using stacked sieves. Clean soil will be provided by the United States Geological Survey (USGS). Unlike the coarseness of quartz sand, clean soil will more accurately approximate the typical grain size and texture of the Libby samples being processed and will reduce the chance of over-processing.

The grinder is adjusted acceptably if, after grinding of the clean soil sample, all material passes through a 60-mesh (250 µm) screen and is substantially retained by a 200-mesh (74 µm) sieve. If a significant amount of the ground clean soil sample is retained on the 60-mesh screen, or if a substantial fraction of the material passes through the 200-mesh screen, adjust the plates of the grinder until these targets are achieved. If the required particle size cannot be achieved even after plate adjustment, other grinder maintenance such as plate replacement may be required. Regardless, grinding of field samples cannot resume until the desired particle size is achieved. Document the grinder number, verification of acceptable adjustment and any observations in the Grinder Calibration and Maintenance Log.

Samples will be weighed following grinding activities. The analytical balance will be calibrated daily with S-1 class weights before processing begins. All measurements, any required maintenance, and the analytical balance number will be recorded in the Analytical Balance Calibration and Maintenance Log.

---

[4] Note that the particle size is cited as "approximately 250 µm". This is due to the nature of grinding asbestos material. Some material that is longer than 250 µm may pass through the grinder if its longest side is parallel with the vertical grinder plates. The material that comes in contact more nearly perpendicular to the vertical grinder plates will be ground to <250 µm

9.2 Grinding Blanks

One grinding blank per grinder will be prepared daily, and will be associated with all samples prepared by that grinder on that day. For further information on grinding blanks refer to Section 12.2.

9.3 Grinding of Fine Field Samples

The sample portion that was sieved to < 1/4 inch will be ground to a particle size of approximately 250 µm. Set up a catch pan under the grinder to collect all the ground material. Take the fine sample set aside in Section 8.2, load the grinder hopper, and allow the fine sample to pass through the plate grinder into the catch pan. Note the technician's initials, date of grinding, and grinder number in the Sample Preparation Log.

The net recovery of fine ground material must not be less than 90% of the mass of fine material placed into the grinder. If recovery is less than 90%, soil grinding must be stopped and the grinder re-adjusted until the mass recovery of test sand and/or soil samples exceeds 90%.

9.4 Decontamination

Plate Grinder

The details of decontamination of the plate grinder and it associated containers and equipment may vary depending on the model of grinder that is being used.

If the plate grinder can be readily disassembled for cleaning without altering its grinding properties, disassemble the grinder and clean the chutes and plates with the HEPA vacuum and compressed air. Then, if needed, use wet wipes to ensure decontamination. If wet wipes are used, the plates and chutes must be thoroughly dried before reassembly. If the grinder is not easily disassembled, clean the grinder with the HEPA vacuum and several blasts of compressed air, paying special attention to areas where dust from the grinding process is known to accumulate (e.g., between the plates and areas adjacent to the catch pan clamps). Then, pass an aliquot of approximately 20 g of quartz sand through the grinder to clean out any residual soil. Discard the quartz sand and re-clean the grinder with the vacuum and another round of high pressure air blasts. After this decontamination procedure, the grinder is ready to process the next sample.

In general, all soil containers, hoppers and catch pans associated with use of the grinder should be decontaminated by using a HEPA vacuum and/or wet wipes, followed by a blast of high pressure air.

Calibration Sieves

The stacked sieves used to calibrate the plate grinder will be decontaminated using a HEPA vacuum and compressed air between calibration uses.

10.0 SPLITTING OF THE FINE GROUND SAMPLE

The fine ground soil sample should be distributed into four approximately equal subsamples using a splitter. All splitting activities will be performed in the hood. Refer to Section 7.1 for details regarding the frequency of ventilation hood calibration.

10.1 Splitting Procedure for Fine Ground Sample

The following method for splitting a soil sample was adapted from EPA 540-R-97-028 (USEPA, 1997):

- Set up one receiving pan on each side of the splitter. Load the soil from the grinder catch pan (Section 9.3) into the splitter, collecting the sample in two receiving pans.
- Tap the catch pan vigorously several times to free any remaining material. Tap the splitter to facilitate the flow of all material through the chutes into the receiving pans.
- Empty one receiving pan into the grinder catch pan and the other receiving pan into the sieve catch pan. Set the sieve catch pan aside; this portion of fine ground sample will be split again later.
- Replace the receiving pans under the splitter. Take the grinder catch pan, containing half of the fine ground sample, and re-load the contents into the splitter as detailed above. Repeat the process of dispersing the sample material by shaking the catch pan and tapping the splitter to uniformly distribute the sample. The resulting splits are the "FG1" and "FG2" portions in the Sample Preparation Log.
- Take these two portions and carefully transfer each into a clean, tared, ziplock sample bag. Re-bag one sample portion in another clean ziplock sample bag and identify this fine ground sample with the Index ID, the suffix "FG" (for "fine fraction, ground") and the fraction number 1, (ex. CS-12345-FG1 for fine ground fraction #1). Identify the bagged second portion with the Index ID, the suffix "FG" and the fraction number 2 and set aside to be re-bagged with the following fine ground portions:

- Place the two empty receiving pans from the "FG1" and "FG2" portion next to the splitter. Repeat the splitting procedure using the other fine ground portion set aside in the sieve pan and split the remaining sample material to create the "FG3" and "FG4" portions.

- Take the remaining "FG3" and "FG4" portions and carefully transfer each into a clean, tared, ziplock sample bag, identify each remaining fine ground sample with the Index ID as noted above.

- Weigh each sample portion (FG1 through FG4), and record each mass along with the technician's initials and date in the Sample Preparation Log.

Combine all of the bagged coarse and fine portions of the sample into one large clean, ziplock sample bag.

Coarse and fine ground samples are now ready to be packaged for shipment to the analytical laboratory or archived as directed. When samples are requested for shipment, the "FG1" fraction will be sent first. If further analyses are required for the fine ground portion, the subsequent fractions will be double bagged and sent (i.e., FG-2 then FG-3, etc.). All archived fine ground portions will be filed in the appropriate inventory archive box noted in the Sample Preparation Log.

10.2 Decontamination

The splitter must be decontaminated between each sample. Use the vacuum and/or wet wipes followed by a blast of compressed air to decontaminate the splitter and brush or wipe off any visible material that is not removed by the vacuum or air blast. The splitter is now ready to process the next sample.

11.0 DOCUMENTATION

Index ID numbers are recorded in the Sample Drying Log, Sample Preparation Log and on all sample containers. Sample Drying Logs and Sample Preparation Logs will be filed or archived according to their associated dry batch and preparation batch number. If revisions to the Sample Drying Log and/or Sample Preparation Log are necessary, the appropriate parties will be notified of the changes, however, these changes will not necessitate revision to the current standard operating procedure, a modification form will be filled out to document the revisions.

As mentioned above, the following equipment calibration and maintenance logs will also be maintained:

- Daily analytical balance calibration using S-1 class weights (Attachment 2)
- Daily grinder setting verification for calibration check and/or post-adjustment verification, grinder maintenance as necessary (Attachment 3)
- Daily ventilation hood operating condition verification (i.e., inline filter checks, changes) (Attachment 4)
- HEPA vacuum maintenance and bag changes (Attachment 5)
- Weekly oven temperature calibration, oven maintenance as necessary (Attachment 6)

In addition, a laboratory notebook will be maintained by each individual or team that is preparing samples. For each day that samples are processed, the following information should be collected:

- Date
- Time
- Personnel
- Personal protective equipment (PPE)
- SOP (including revision number) and any other laboratory-specific governing plan being followed
- Descriptions of any deviations to the SOP, the reason for the deviation and/or any modification forms being followed
- Summary of laboratory activities (including number of samples prepared, and equipment calibrated and used)

12.0 QUALITY CONTROL

Quality control (QC) samples are inserted into the sample train to monitor for potential contamination introduced during the preparation process or to assess accuracy of analysis that may be affected due to preparation procedures. If samples results indicate the occurrence of contamination or inconsistent results, the PL^2 will be notified. The PL^2 will then notify the EPA Regional Project Manager and the Regional Chemist in order to review laboratory procedures and identify any changes in preparation laboratory methods and procedures that may be necessary. Any such reviews and resultant changes will be documented accordingly by the PL^2.

12.1 Preparation Blanks

A preparation blank is a sample of 200-400 grams of clean quartz sand that is treated identically to a field soil sample. That is, the preparation sample is dried in the oven along with the field soil samples, split into archive and preparation fractions using a riffle splitter, screened through a ¼ inch screen (even though there are no particles larger than ¼ inch), and ground by passing through the plate grinder. This type of sample is intended to detect contamination that may occur at any stage of the soil preparation procedure.

At least one preparation blank will be processed with each drying batch of approximately 20 field samples. Preparation blanks will be assigned a random and unique Index ID and will be submitted to the laboratory blind. The Index ID assigned to each preparation blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any preparation blanks at a level greater than Non-detect (Bin A) by PLM-VE should be taken as a sign of potential cross-contamination, and all field samples associated with the preparation batch for the preparation blank having detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any of the corresponding field samples. If the overall fraction of preparation blanks that contains detectable asbestos (> Bin A) exceeds 1%, a review of laboratory procedures should be undertaken to identify and address the source of the contamination.

12.2 Grinding Blanks

A grinding blank consists of 100-200 grams of clean quartz sand that is passed through the plate grinder. The purpose of this type of sample is to evaluate the effectiveness of decontamination procedures for the plate grinder.

One grinding blank per grinder will be prepared for each day that field samples are being ground. Each grinder used in the laboratory will be assigned a number and all samples processed will be associated with the grinder used for preparation. The grinder number used for each sample will be noted in the Sample Preparation Log. Grinding blanks will not be dried, split for archive, or sieved. Rather, a grinding blank will only be ground and split into four fine ground samples. The grinding blank is assigned a random and unique Index ID and is submitted to the laboratory blind. The Index ID assigned to each grinding blank must be in accord with the numbering system specified in the program-specific project plan.

Detection of asbestos fibers (any type) in any grinding blank at a level greater than Non-detect (Bin A) should be taken as a sign of potential cross-contamination, and all field samples associated with the grinding blank that reports detectable asbestos (> Bin A) will be reviewed and qualified appropriately if detectable levels of asbestos are also found in any pf the corresponding field samples. If the overall fraction of grinding blanks that contains detectable asbestos (> Bin A) in a soil preparation facility exceeds 1%, steps should be taken to develop an improved method for grinder decontamination.

12.3 Performance Evaluation Samples

Performance Evaluation (PE) samples are samples of Libby soil that have been spiked with a known amount of Libby Amphibole (LA) asbestos. These samples were prepared by the USGS

for use at the Libby site by spiking uncontaminated soil from Libby with a known mass of LA fibers collected at the mine site, and then grinding the sample to a particle size of ≤ 250 um as described above. Several different concentration values of PE samples were prepared, ranging from < 0.1% to 2%. Each bottle contains about 100 grams of the PE material.

PE samples will be utilized in two ways.

First, the soil preparation facility will insert untreated PE samples into the analytical sample train sent to the laboratory for PLM-VE analysis. This type of PE sample is intended to evaluate the performance of the analytical laboratory (rather than the preparation facility).

Second, the soil preparation laboratory will process PE samples in the same way that field soil samples are processed, as detailed below. This type of PE sample is intended to determine if there is any loss of asbestos during sample processing. In addition, considered in conjunction with a grinding blank that is passed through the decontaminated grinder immediately following the PE sample, the PE sample will also be used to facilitate assessment of grinder decontamination procedures.

The frequency of each type of PE sample (unprocessed and processed) should be one per month for each month in which soil processing is occurring. These should be distributed approximately evenly between the different concentration values that are available for PE samples.

Each month that soil processing is occurring, the procedure to be followed for generation and submittal of PE samples is as follows:

1. Select a PE bottle for inclusion.
2. Thorough mix the contents of the PE bottle by inversion (a minimum of 10 times) and/or rolling (a minimum of 10 minutes).
3. Remove an aliquot of about 20 grams and package this for submission to the analytical laboratory without any processing. If more than one laboratory is analyzing samples, rotate the submittal of unprocessed samples so that all laboratories receive approximately equal total number of unprocessed PE samples.
4. Take the remainder of the PE bottle (about 80 grams) and carry this material through the full sequence of steps applied to each field sample, starting with oven drying. After splitting the dried sample with the riffle splitter, recombine the samples so that the full 80 grams is screened through the ¼ inch sieve and passed through the plate grinder. Thus, there is no archive split for PE samples. After grinding and splitting, this should result in four sub-samples of processed PE sample. Prepare three of these for submittal to the analytical laboratories, and hold one sample in archive.

Results of PE samples processed by the soil preparation laboratory are evaluated by comparing the reported results for LA to the nominal results. Deviations from nominal may be the result of variations either in soil processing procedures and/or in the analytical procedure. If the frequency of strongly discordant results (i.e., the results of the PE sample differ by more than one bin from the nominal result) exceeds 10%, then the source of the inconstancy should be investigated and remedied.

12.4 Preparation Duplicates

A preparation duplicate is prepared by using a riffle splitter to divide a field soil sample into two approximately equal portions, creating a parent and duplicate sample. Both samples are then processed in the same fashion. The preparation duplicate is assigned a unique Index ID, and is submitted to the laboratory blind. The Index ID assigned to each preparation duplicate must be in accord with the numbering system specified in the program-specific project plan.

One preparation duplicate sample will be processed for every 20 field samples prepared (5%). Results from duplicate samples serve to evaluate the precision of the combined sample preparation process and the laboratory analysis. Inconsistent results between parent and duplicate may be due either to variability in sample preparation, sample analysis, and/or to small scale variability in the sample that is not fully controlled by mixing and splitting. If the overall frequency of strongly discordant results (i.e., the results for the parent sample and duplicate are different by more than one bin) is greater than 10%, steps should be taken to identify and address the source of the variability in the sample preparation procedure.

13.0 DECONTAMINATION

All non-disposable equipment used during soil sample preparation must be decontaminated prior to use. Scoops, spoons, splitters, sieves and drying pans that are re-used must be decontaminated with a HEPA vacuum, compressed air, wet-wiping and/or by brushing off any residual material. If soil particles are visible on any of the equipment, repeat the decontamination procedure until the equipment is clean. To reduce the potential for human exposure in the laboratory, COMPRESSED AIR SHOULD BE USED CAREFULLY AND ONLY UNDER VENTED HOODS.

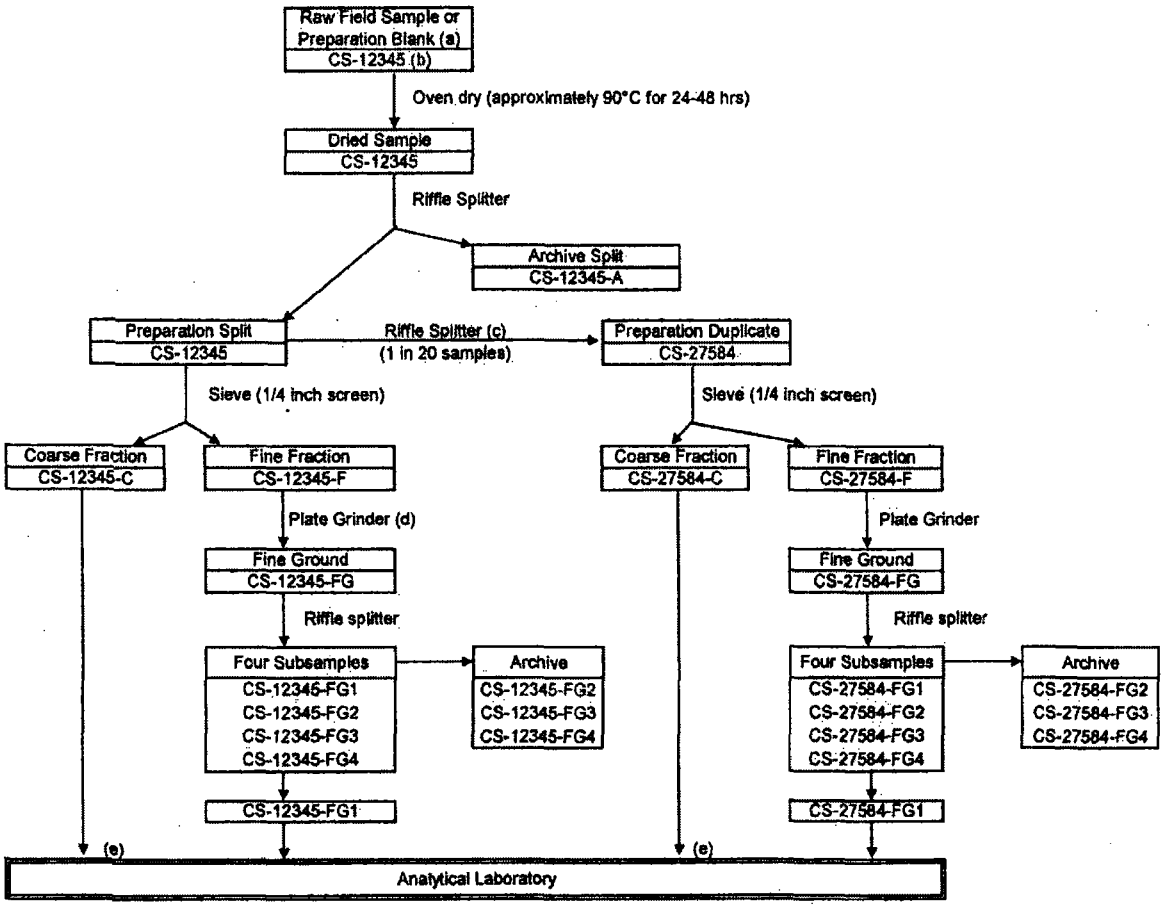
Detailed decontamination procedures for specific equipment are noted in Sections 6.3, 7.5, 8.3, 9.4, and 10.2.

14.0 REFERENCES

American Society for Testing and Materials. 1998. Standard Practice for Reducing Samples of Aggregate to Testing Size, ASTM Designation: C 702 - 98, 4 p.

USEPA. 1997. Superfund Method for the Determination of Releasable Asbestos in Soils and Bulk Materials. EPA 540-R-97-028.

FIGURE 1 SOIL PREPARATION FLOW DIAGRAM



NOTES:

(a) A preparation blank (200-400 grams of clean silica sand) is prepared in the same way as field samples at a rate of 5%

(b) Example Index ID (sample number) shown to illustrate naming conventions

(c) A preparation duplicate is prepared at a rate of 5%

(d) A grinding blank (100-200 grams of clean sand) is passed through the plate grinder and split into 4 sub-samples at a rate of 5%

(e) Coarse sample will be returned to EPA for archive after laboratory analysis

ATTACHMENT 1

SAMPLE DRYING AND SAMPLE PREPARATION LOG SHEETS

Sample Drying Log Sheet

Laboratory Name: _____ Sheet No.: _____

Drying Begun: date _____ time _____

Drying Complete: date _____ time _____

Oven number: _____

Oven temp: _____ °C

	Index ID	Inventory ID No.	SOP and Rev No.	Sample mass (g)			Original Sample ID and Notes (indicate if preparation blank)	QC
				Before drying	After Drying	Initials and date		Initials and Date
1								
2								
3								
4								
5								
6								
7								
8								
9								
10								
11								
12								
13								
14								
15								
16								
17								
18								
19								
20								
21								
22								
23								
24								
25								

Sample Preparation Log Sheet

Laboratory Name: _____ Sheet No.: _____

Preparation Batch: _____

	Index ID	SOP and Rev No.	Inventory ID	Drying Batch ID	Archive Sample Splitting	Duplicate Sample Splitting	Sieving			Sample Grinding		Sample Splitting					Original Sample Identification and Notes (indicate if grind blank, prep blank, or duplicate pair. For duplicate pair enter the parent ID)	QC
							Sample Mass (g)					Sample Mass (g)						
					Initials and date	Initials and date	Coarse Fraction > 1/4"	Fine Fraction < 1/4"	Initials and Date	Initials and Date	Grinder #	FG1	FG2	FG3	FG4	Initials and Date		Initials and Date
1																		
2																		
3																		
4																		
5																		
6																		
7																		
8																		
9																		
10																		
11																		
12																		
13																		
14																		
15																		
16																		
17																		
18																		
19																		
20																		

The following preparation steps require Technician Initials and Date to document activity: Sample Drying, Archive Sample Splitting, Preparation Duplicate Splitting, Sieving, Homogenization, Sample Splitting

ATTACHMENT 2

ANALYTICAL BALANCE CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = _____

Balance # = _____

Measurement Number		S - 1 Class Weight Measurements				Measurement within range? Yes or No	If "No" Recalibrate	Technician Initials	QC check initials
	Calibration Weights	0.1 g	1 g	10 g	100 g				
	Tolerance Limit Range	0.05 - 0.15 g	0.90 - 1.10 g	9.75 - 10.25 g	99.00 - 101.00 g				
	Date								
1									
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									

The analytical balance calibration will be verified daily.
All tolerance limits are standard tolerance limits for Class S-1 weights.
After 20 measurements, the tolerance range will be evaluated for reasonableness.
Weights falling outside the range require that the balance be recalibrated using all S-class weights

Sheet No.: Balance - _____

ATTACHMENT 3

GRINDER CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = _____

Grinder # = _____

Date	Activity Type (D, A, M)	Sieve Test: 60 mesh acceptable Y or N	Sieve Test: 200 Mesh acceptable Y or N	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

D = daily calibration
A = Adjustment Calibration Verification
M = Maintenance Only
For the 60 mesh sieve test to be acceptable all of the clean soil must pass through the sieve.
For the 200 mesh sieve test to be acceptable a substantial portion of the ground soil must be retained on the sieve.
Failure of either sieve test requires adjustment of the plates followed by adjustment verification prior to grinding samples.

Sheet No.: Grinder - _____

ATTACHMENT 4

VENTILATION HOOD CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = ______________________________

Ventilation Hood # = _________________

Date	In-line HEPA indicators acceptable?	Notes (Include description of action/maintenance)	Technician Initials	QC Check Initials

Ventilation system operating conditions should be verified daily

In-line system should indicate the velocity/negative flow is 100 ft/min. If not, the problems with the system must be corrected prior to processing samples. Note actions or maintenance taken.

Sheet No.: Hood - ______________

ATTACHMENT 5

HEPA VACUUM CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = ______________________

Vacuum # = ____________

Date	System Check Acceptable? Yes or No	HEPA filter clean? Yes or No	Bag fill level acceptable? Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Physically check the vacuum suction and note audible change in motor daily. If significant reduction in the vacuum suction or motor "strain" is audibly noted then the system check is unacceptable. If unacceptable, perform and document the HEPA filter and bag maintenance checks above and perform maintenance.

Sheet No.: Vacuum - ____________

ATTACHMENT 6

OVEN CALIBRATION AND MAINTAINANCE LOG SHEET

Preparation Laboratory = _____

Oven # = _____

Date	Certified Temperature Reading (°C)	Thermometer ID	System Check Acceptable? (90+/-1 °C) Yes or No	Notes (Include description of action/maintenance performed)	Technician Initials	QC Check Initials

Sheet No.:Oven - _____

LIBBY SUPERFUND SITE STANDARD OPERATING PROCEDURE

APPROVED FOR USE IN LIBBY SUPERFUND SITE ONLY

Date: April 21, 2004 SOP No. SRC-LIBBY-01 (Rev. 2)

Title: QUALITATIVE ESTIMATION OF ASBESTOS IN COARSE SOIL BY VISUAL EXAMINATION USING STEREOMICROSCOPY AND POLARIZED LIGHT MICROSCOPY

Author Sally M. L. Gibson Syracuse Research Corporation

SYNOPSIS: A standardized method is described for the examination of the coarse fraction (>1/4") of soil samples using stereomicroscopy and polarized light microscopy (PLM) to identify, segregate, and estimate the mass percent of asbestos in the sample matrix.

Received by QA Unit:

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Region 8	Mary Goldade	4/21/04
Syracuse Research Corp.	W S Brothers	4/26/04

Revision	Date	Reason for Revision
0	11/12/02	--
1	5/20/03	Provided clarification on dealing with very small particles.
2	4/21/04	Included statements on limitations of intended use

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized screening method for the visual examination of the coarse fraction of previously sieved soil samples for evidence of asbestos mineral content using stereomicroscopy with confirmation of asbestos content by polarized light microscopy (PLM). This SOP incorporates salient components of EPA Test Method 600/R-93/116 *Method for Determination of Asbestos in Bulk Building Materials* and National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM*, Issue 2.

This procedure will be used by employees of contractors/subcontractors supporting USEPA Region 8 projects and tasks for the Libby, Montana, site. Deviations from the procedure outlined in this document must be approved by the USEPA Region 8 Remedial Project Manager or Regional Chemist prior to initiation of sample analysis.

2.0 PREREQUISITE TRAINING

Visual examination will be performed according to this SOP by a laboratory accredited by the National Voluntary Laboratory Accreditation Program (NVLAP) and by analysts proficient either by education or experience in asbestos mineral identification by stereomicroscopy and PLM. Analyst familiarity with the procedural applications prescribed in EPA Test Method 600/R-93/116 and NIOSH Method 9002 is required.

Training as described in the Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4, (CSS SQAPP [CDM 2002]) will be provided to laboratory personnel or laboratories with less than one year of project-specific experience by "mentors" from either Reservoir Environmental Services, Inc. or EMSL.

3.0 RESPONSIBILITIES

The CDM Laboratory Coordinator (LC) is responsible for overseeing the activities of the CDM Soil Preparation Laboratory and subcontracted laboratories performing sample analysis for the Libby, Montana, project. The LC is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the CSS SQAPP. It

is the responsibility of the LC to communicate with the project personnel and subcontracted laboratory regarding specific analysis objectives and anticipated situations that require any deviation from the CSS SQAPP SOPs. In addition, it is the responsibility of the LC to communicate the need for any deviations from this SOP with the CDM Project Manager, USEPA Region 8 personnel (Remedial Project Manager or Regional Chemist.)

Subcontracted laboratory analysts performing the visual examination are responsible for adhering to the applicable tasks outlined in this SOP and substantiating components of the reference procedures (EPA 1993; NIOSH 1994) with the modifications contained herein.

4.0 EQUIPMENT

- Analytical balance - accurate to 0.01 g, range of 0.01 g to 1000 g (for weighing total sample)
- Analytical balance - accurate to 1 mg (for weighing asbestos)
- Traceable standards - major asbestos types
- Microscope - binocular stereomicroscope, 5-60X approximate magnification
- Microscope - polarized light, binocular or monocular with a cross hair reticle (or functional equivalent) and magnification of at least 8X
  - 10X, 20X, and 40X objectives
  - 360 degree rotatable stage
  - substage condenser with iris diaphragm
  - polarizer and analyzer which can be placed at 90 degrees to one another and calibrated relative to the cross-line reticle in the ocular
  - port for wave plates and compensators
  - wave retardation plate (Red I Compensator) with ~550 nanometer retardation and known slow and fast vibration directions
- Light Sources - incandescent or fluorescent

- Tweezers, dissecting needles, scalpels, probes, razor knives, etc. - standard sample manipulation instruments/tools
- Microscope slides and cover slips
- Refractive index liquids
- Pre-tared glassine paper, glass plates, weigh boats, petri dishes, watchglasses, etc. - laboratory sample containers
- HEPA-filtered or Class 1 biohazard hood negative pressure
- Three-ring binder book- binders will contain Microscopic Examination Logbook Sheets (Attachment 1)

5.0 METHOD

Soils from the Libby, Montana site will be dried, sieved, and prepared according to the most recent revision of SOP ISSI-LIBBY-01, Soil Sample Preparation. The coarse fraction of the soil sample is defined as that portion of the sample which does not pass through a 1/4" sieve. The coarse fraction will be weighed, placed in a zip-top plastic bag, and labeled as described in Camp, Dresser, and McKee (CDM) SOP 1-3 (with project-specific modifications). The samples will be packaged and shipped by the soil preparation laboratory as described in CDM SOP 2-1 (with project-specific modifications) and transferred to the laboratory via chain-of-custody procedures described in CDM SOP 1-2 (with project-specific modifications).

The following sections describe the stereomicroscopic and PLM examination. Materials tentatively characterized as asbestos by stereomicroscopy will be isolated and subjected to confirmation by PLM. The mass % of Libby amphibole asbestos, other amphibole asbestos, and chrysotile asbestos in the coarse soil fraction will be calculated from the mass of each asbestos type positively identified by PLM and the original sample weight. Figure 1 provides an overview of the process.

5.1 Stereomicroscopic Examination

The laboratory will receive the coarse fraction soil samples from the CDM Soil Preparation Laboratory. The entire sample will be weighed and placed in an appropriate container. The weight of each coarse sample will be recorded, along with the sample identification, on the Microscope Examination Logbook Sheet. The sample will be subject to stereomicroscopic examination and particle segregation as depicted Figure 1. The stereomicroscopic examination to identify and segregate asbestos includes:

- using multiple fields of view over the entire sample
- probing the sample by turning pieces over and breaking clumps where possible
- manipulating the sample using appropriate instruments/tools
- observing homogeneity, texture, friability, color and extent of any observed asbestos in the sample(s)

NOTE: Although the coarse fraction is prepared by sieving with a 1/4" screen, particles smaller than 1/4" may be present in the fraction due to adherence between coarse and fine particles. This may even include some very fine asbestos fibers. Because of the technical difficulty, the analyst should not attempt to physically segregate and weigh particles smaller than about 2-3 mm (1/10 inch). A particle this size is expected to have a mass of about 10-20 mg, which is less than 0.1% of a sample whose total mass is 25 grams. If no particles larger than 2-3 mm are present, this should be noted in the data sheet for each category of asbestos using the following code system:

- ND = No asbestos observed
- Tr = Trace levels of asbestos observed but not quantified

The weight fraction for any asbestos type marked "ND" or "Tr" in a given sample is not calculated and is left blank.

As the sample is examined, the analyst will continue segregation of the sample until the entire coarse soil fraction has been characterized as either "non-asbestos" or "tentatively identified asbestos." The tentatively identified asbestos particles will be examined by PLM, as described below. The stereomicroscopist will initial and date the Microscopy Examination Logbook Sheet.

5.2 PLM

The coarse material tentatively identified as asbestos by stereomicroscopic examination will be subject to confirmation using PLM, as described in SOP SRC-LIBBY-03 (Revision 0) ("Analysis of Asbestos Fibers in Soil by Polarized Light Microscopy"). The PLM examination will be used to confirm that the particles tentatively classified as asbestos are actually asbestos, and will be assign each particles to one of three categories:

LA = Libby amphibole
OA = Other amphibole
C = Chrysotile

If OA is observed, the type of OA observed should be noted in the data sheet using the following code system:

- AMOS = Amosite
- ANTH = Anthophyllite
- CROC = Crocidolite
- UNK = Unknown

The total weight of each type of positively identified asbestos (LA, OA, C) will be determined and recorded on the Microscopic Examination Logbook Sheet, along with the analyst's initials and the date of the examination.

6.0 QUALITY ASSURANCE

Laboratories performing the examination must be accredited by NVLAP. "Calibration" should be verifiable for each microscopist in terms of project-specific training and the successful analysis of materials of known asbestos content (NVLAP test samples, in-house standards) similar to those anticipated to be observed in Libby, Montana soils. Additionally, references such as photographs of the asbestos minerals illustrating distinguishing properties should be available benchside during characterization.

Quality control samples as described in ISSI-LIBBY-01 (i.e., preparation duplicates) will not submitted for the coarse materials samples. The entire coarse fraction will be subject to examination.

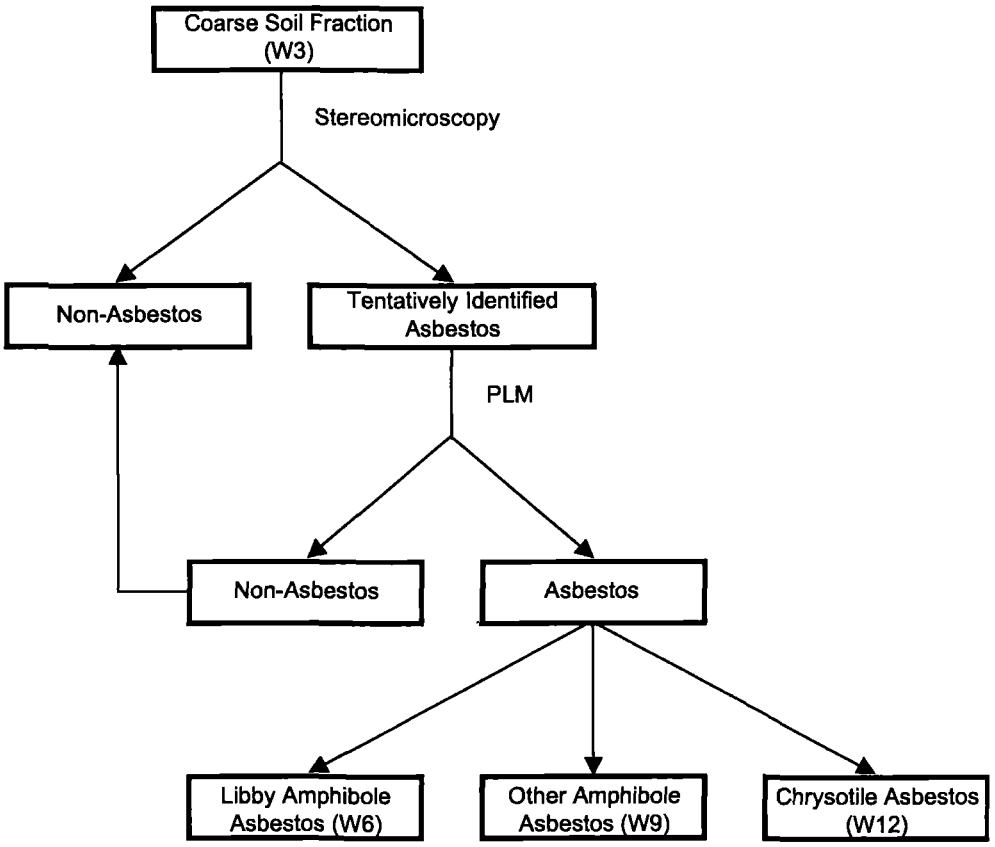
7.0 REFERENCES

CDM 2002. *Sampling and Analysis Plan, Remedial Investigation, Contaminant Screening Study, Libby Asbestos Site, Operable Unit 4.* 3282-116-PP-SAMP-14187. Camp, Dresser and McKee Denver, Colorado. April.

NIOSH 1994. National Institute of Occupational Safety and Health (NIOSH) Method 9002 *Asbestos (bulk) by PLM,* Issue 2.

USEPA 1993. *Method for Determination of Asbestos in Bulk Building Materials.* 600/R-93/116.

Figure 1. Overview of Sample Examination Process



W3 = Original coarse soil fraction mass (g)
W6 = If present in measurable quantities, mass (mg) of Libby amphibole
W9 = If present in measurable quantities, mass (mg) of other amphibole
W12 = If present in measurable quantities, mass (mg) of chrysotile

Codes used in the illustration (e.g., W3) correspond to Data Log Sheet

ATTACHMENT 1

MICROSCOPIC EXAMINATION LOGBOOK SHEET

SRC-LIBBY-01 Data sheet and EDD.xls
(Check with Volpe or SRC to determine the latest version number)

Example hard copy of data entry sheet shown on next page (for illustration purposes only).

Data Log Sheet v6 for SOP SRC-LIBBY-01

Stereomicroscopic and Gravimetric Analysis of Coarse Soil

Lab Name:

SOP Version:

Lab Job No.

Page _____ of _____

Calculated automatically in the "Electronic Data Entry" form. Do not enter data here.

EPA Index ID	Index Suffix	Lab Job-Sample No.	Status (1 = Analyzed, 2 = Missing, 3 = Contam, 4 = Cancelled)	QA Type (Not QA)	Total Sample Weight (g)			Analysis Details		Mass of Asbestos Particles (mg)																Comments (see Notes below)
										Libby Amphibole (LA) Asbestos				Other Amphibole (OA) Asbestos					Chrysotile (C) Asbestos							
					Tare Weight (g) Empty Container	Mass of Sample + Container (g)	Mass of Sample (g)	Analyst Initials	Analysis Date	LA Qual* (ND, Tr)	Tare Weight - Container (mg)	Mass of LA + Container (mg)	Mass (mg) LA	OA Qual* (ND, Tr)	OA Type** (AMOS, ANTH, CROC, UNK)	Tare Weight - Container (mg)	Mass of OA + Container (mg)	Mass (mg) OA	C Qual* (ND, Tr)	Tare Weight - Container (mg)	Mass of C + Container (mg)	Mass (mg) C	% LA	% OA	% C	

Notes:

*Qualifier codes: ND = No asbestos observed.
Tr = Trace levels observed but not quantified.

**OA Type codes: AMOS = Amosite
ANTH = Anthophyllite
CROC = Crocidolite
UNK = Unknown

Comment Codes (user-defined):

ANALYSIS OF ASBESTOS FIBERS IN SOIL BY POLARIZED LIGHT MICROSCOPY

SYNOPSIS: A semi-quantitative method for identifying and quantifying asbestos fibers in soil using polarized light microscopy is provided. This method is based on NIOSH Method 9002, EPA Method 600/R-93/116, and CARB Method 435, with project specific modifications intended specifically for application at the Libby Superfund Site. Sampling and plan developers and data users are cautioned to understand how data are generated from this SOP.

APPROVALS:

USEPA Region 8

Signature _____ Date 10/10/08

Mary Goldade
Print Name

Senior Environmental Scientist/Chemist
Title

ESAT Region 8

Signature _____ Date 10/10/08

JOHN CALONNS
Print Name

ESAT TEAM MANAGER
Title

Revision	Date	Principal Changes
0	3/3/2003	Initial Author: William Brattin (Syracuse Research Corporation)
1	12/11/2003	Clarified binning assignment of samples at 0.2%. Author: William Brattin (Syracuse Research Corporation)
2	10/10/2008	Complete re-design of the SOP. Provided specific requirements for sample preparation and analytical process. Authors: Douglas Kent and Nikki MacDonald, ESAT Region 8

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1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standard approach for semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by Polarized Light Microscopy (PLM). This SOP is specifically intended for application at the Libby Asbestos Superfund Site and has been refined to focus testing on Libby Amphibole asbestos at levels below 1%.

2.0 SCOPE AND APPLICATION

This method is intended for analysis of asbestos in soil or other similar soil-like media in which the soil has been taken through a preparation process described below. This method is appropriate for the analysis of all types of asbestos fibers (chrysotile and amphiboles), including those that are characteristic of the Libby Asbestos Superfund Site, Libby Amphibole asbestos (LA).

3.0 RESPONSIBILITIES

3.1 It is the responsibility of the laboratory supervisor to ensure that all analyses and quality assurance (QA) procedures are performed in accordance with this SOP, and to identify and take appropriate corrective action to address any deviations that may occur during sample preparation or analysis.

3.2 The Laboratory Manager, QA/QC Coordinator (or equivalent), or Analytical Lead communicates with project managers at the United States Environmental Protection Agency ([EPA]; also referred to as the client), or their designate, any situations where a change from the SOP may be useful and/or required. The laboratory supervisor must receive approval from the EPA for any deviation or modification from the SOP before incorporating any such deviation or modification into the sample preparation and analysis process (Refer also to Section 8.2).

3.3 It is the responsibility of the laboratory to maintain a PLM SOP for Bulk Asbestos Materials, Quality Assurance Manual (QAM), Quality Management Plan (QMP), or an equivalent document(s) that meets all the requirements of the National Voluntary Laboratory Accreditation Program (NVLAP) Handbook 150. It is also the responsibility of the laboratory to ensure its testing activities stay in compliance with the requirements of NVLAP Handbook 150 and the regulatory and accrediting agencies that provide oversight of the laboratory's operations and all Libby Asbestos Site project-specific requirements.

4.0 METHOD DESCRIPTION

4.1 The test method describes a semi-quantitative analysis of asbestos in samples of soil or other soil-like materials using the visual area estimation technique by PLM, referred to as PLM-VE. The test method used for analyzing PLM asbestos samples specific to the Libby Asbestos Superfund Site is based on the National Institute of Occupational Safety and Health (NIOSH) Method 9002, EPA Method 600/R-93/116, and the State of California Air Resources Board (CARB) Method 435, with project-specific modifications provided in this SOP.

4.2 Soil samples for the Libby project are processed according to the current version of SOP ISSI-LIBBY-01, Soil Sample Preparation, before submittal to the laboratory for analysis. This process separates the coarse fraction of the soil from the fine fraction (particles passing through a ¼ inch sieve). The fine fraction is homogenized and ground to a maximum particle size of approximately 250 microns (μm). This fine fraction is further sub-divided into four fractions using a riffle splitter. One or more of these fractions is then submitted to an approved and accredited PLM laboratory for analysis. This SOP is specific to only the analysis of the fine fractions of soil samples. Coarse fractions of soil samples are analyzed according to the current version of SOP SRC-LIBBY-01, Qualitative Estimation of Asbestos in Coarse Soil by Visual Examination Using Stereomicroscopy and Polarized Light Microscopy.

4.3 The fine fraction soil sample to be evaluated for asbestos content is first examined using a low magnification stereomicroscope. Microscope slide mounts are then prepared of the sample by immersing sample material in a liquid medium of known refractive index (RI). These slide mounts are then analyzed visually by PLM. Asbestos and non-asbestos phases are identified on the basis of their morphology and optical properties. Quantification of the amount of asbestos present is done using a visual estimation approach. The concentration of LA in the sample is estimated in terms of mass fraction (percent asbestos by weight) based on the use of project-specific reference materials. Samples are re-analyzed or re-prepped and re-analyzed, and prepared standards are analyzed, as part of the quality control (QC) program.

5.0 ACRONYMS

ACM	Asbestos Containing Material
CARB	State of California Air Resources Board
EDD	Electronic Data Deliverable
EDS	Energy Dispersive Spectrometry
EDXA	Energy Dispersive X-ray Analysis
EPA	United States Environmental Protection Agency
HEPA	High Efficiency Particulate Air
LA	Libby Amphibole asbestos
LDC	Laboratory Duplicate – Cross-check
LDS	Laboratory Duplicate – Self-check
LIMS	Laboratory Information Management System
MSDS	Material Safety Data Sheet
NIOSH	National Institute for Occupational Safety and Health
NIST	National Institute of Standards and Technology
NVLAP	National Voluntary Laboratory Accreditation Program
PE	Performance Evaluation
PLM	Polarized Light Microscopy
PLM-VE	Visual Area Estimation technique employed by Polarized Light Microscopy
PPE	Personal Protective Equipment
QA	Quality Assurance
QAM	Quality Assurance Manual
QC	Quality Control
QMP	Quality Management Plan
RI	Refractive Index
SEM	Scanning Electron Microscopy

SOP	Standard Operating Procedure
SRM	Standard Reference Material
TEM	Transmission Electron Microscopy
µm	Microns (1,000 µm = 1mm)
USGS	United States Geological Survey

6.0 HEALTH AND SAFETY

6.1 Follow general laboratory health and safety policies and regulations in the laboratory's Health and Safety Plan, Chemical Hygiene Plan, or equivalent.

6.2 All sample handling and preparation activities must be performed in a ventilated hood with an operating High Efficiency Particulate Air (HEPA) filtration system, a class 1 biohazard hood, or glove box with continuous airflow (negative pressure). Never have a sample container open except when the sample is inside of the sample preparation hood. Appropriate personal protective equipment (PPE) should be worn at all times.

6.3 Avoid repeated or prolonged contact with the RI liquids and inhalation of fumes from the RI liquids. Refer to the Material Safety Data Sheet (MSDS) forms for RI liquids for additional information and cautions.

7.0 CAUTIONS

7.1 The toxicity or carcinogenicity of each reagent (e.g., RI liquids) used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be avoided.

7.2 After processing each sample, use distilled water and paper towels to thoroughly decontaminate all work surfaces and utensils that came into contact with a sample and/or RI liquid. Never have more than one sample container open at any one time.

8.0 GENERAL LABORATORY PRACTICES

8.1 QA Program

8.1.1 Each laboratory operates under a QA program appropriate to the type, range, and volume of work it performs.

8.1.2 It is the responsibility of the laboratory to maintain a Quality Management Plan, or equivalent, in which the laboratory's QA program is detailed. Additional QA/QC requirements specific to the PLM laboratory and the Libby project are described later in Section 16.0.

8.1.3 All work is performed at a permanent laboratory location. Even if a laboratory is part of a larger organization, it is able to carry out all testing, calibration, and daily QA/QC activities independently, and at one location. There are no remote or sub-facilities where testing work is performed.

8.2 Documenting SOP Modifications

8.2.1 Any deviation from the SOP shall be documented in a laboratory modification form and then addressed in the technical Case Narrative prepared as part of the test

report.

8.2.2 Additionally, when there is reason to suspect a departure from the SOP has affected the result or certainty of a measurement provided to the client, the client must be notified and informed of the nature of the departure from the SOP and the possible effect on the result or validity of the analysis. The course of action taken to keep the departure from recurring must also be discussed with the client.

9.0 PERSONNEL QUALIFICATIONS

9.1 The use of this SOP is limited to microscopists knowledgeable in the production and evaluation of asbestos data.

9.1.1 All personnel analyzing samples for the Libby project are expected to be familiar with routine chemical laboratory procedures, principles of optical mineralogy, and proficient in EPA Method 600/R-93/116, NIOSH Method 9002, and CARB Method 435.

9.1.2 Personnel at laboratories with less than one year of experience specific to the Libby Asbestos project are required to participate in the laboratory "mentoring" program to obtain additional guidance and instruction. This training is provided by personnel familiar with the particular problems and types of asbestos encountered at the Libby Asbestos Superfund Site.

9.2 Before performing any analyses, the analyst must demonstrate the ability to generate acceptable accuracy and precision with this method. This includes successfully completing NVLAP proficiency testing.

10.0 EQUIPMENT

10.1 The laboratory has all items of equipment (including instrumentation, hardware, software, and reference materials) required for the correct performance of calibrations and tests.

10.2 All equipment is properly maintained and calibrated (as appropriate) prior to use. See Section 12 for further details regarding microscope calibration.

10.3 Following is a general list of the equipment available at the PLM laboratory to perform this SOP:

10.3.1 Polarized Light Microscope, with:

- 10.3.1.1 Light source and replacement bulbs
- 10.3.1.2 Binocular observation tube
- 10.3.1.3 Blue daylight filter
- 10.3.1.4 Oculars (10X)
- 10.3.1.5 Objectives: 10X, 20X, and 40X (or similar magnification)
- 10.3.1.6 10X Dispersion Staining Objective
- 10.3.1.7 360 degree rotatable and centerable stage
- 10.3.1.8 Polarizer and analyzer aligned at 90 degrees to one another
- 10.3.1.9 Bertrand lens (optional)
- 10.3.1.10 Substage condenser with iris diaphragm
- 10.3.1.11 Accessory slot for compensator plate
- 10.3.1.12 First order red (550 nanometer) compensator plate

10.3.1.13 Crosshair reticle
10.3.1.14 Adjustment tools
10.3.2 HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure)
10.3.3 Binocular stereomicroscope, 10-50X magnification (approximate)
10.3.4 Light source for stereomicroscope
10.3.5 Muffle furnace
10.3.6 Analytical balance
10.3.7 SOP-specific Electronic Data Deliverable (EDD), most recent version
10.3.8 Mortars (agate or porcelain)
10.3.9 Pestles (agate or porcelain)
10.3.10 Anemometer
10.3.11 Wet/dry vacuum with HEPA filtration
10.3.12 Decontamination equipment (e.g. baby wipes, wet mop with bucket, etc.)

11.0 STANDARDS, REAGENTS AND SUPPLIES

11.1 High Dispersion RI Liquid from 1.620 to 1.640 (1.625 is a common choice)

11.2 1.550 High Dispersion RI Liquid

11.3 1.680 to 1.700 RI Liquid

11.4 Solid RI Standards (precision optical glass, RI from 1.48 to 1.72, in gradations of 0.01, 25 standards)

11.5 National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 1866b - Common Commercial Asbestos consisting of chrysotile, amosite, and crocidolite

11.6 NIST SRM 1867a - Uncommon Commercial Asbestos consisting of tremolite, amosite, and anthophyllite

11.7 Controlled Performance Evaluation (PE) Reference Materials (prepared for EPA by United States Geological Survey [USGS])

11.7.1 Soils containing LA in various concentrations (provided by the client)
11.7.2 Permanently mounted slides containing 0.2% LA by mass
11.7.3 Permanently mounted slides containing 1.0% LA by mass

11.8 Controlled Libby Amphibole Asbestos (prepared for EPA by USGS), a finely-milled composite of a selected subset of 30 samples taken from the mine at the Libby Asbestos Superfund Site

11.9 NIST Bulk Asbestos Proficiency Testing Round M12001, Sample 4, a sample of un-milled rock-form winchite/richterite taken from the mine at the Libby Asbestos Superfund Site.

11.10 Non-asbestos reference materials (gypsum, calcite, fiberglass, etc.)

11.11 Instrument maintenance/calibration logbooks, document controlled

11.12 RI liquid calibration logbook, document controlled

11.13 Data recording sheet or bench sheet (Attachment 1)

11.14 RI liquid calibration conversion tables (Attachment 2)

11.15 Thermometer, NIST Traceable

11.16 Permanently mounted test slides of Anthophyllite (or other orthorhombic mineral), or the synthetic fiber polypropylene, for alignment of microscope's polars and crosshairs

11.17 Thin section of biotite for alignment of microscope's lower polar (recommended but not required)

11.18 Calibration Standards (see Sections 16.2 and 16.3)

11.19 Glass microscope slides and cover slips

11.20 Slide trays

11.21 Sampling utensils (tweezers, dissecting needles, scalpels, probes, etc.) for sample manipulation

11.22 Clean, asbestos-free sample containers (ceramic evaporating dishes, foil weighing dishes, watchglasses, etc.)

11.23 Aluminum ashing tins

11.24 Distilled water in spray bottles

11.25 Plastic re-sealable sample bags (4 mil poly bags)

11.26 Asbestos Containing Material (ACM) disposal bags

11.27 Crucible tongs

11.28 Autoclave gloves

11.29 Disposable examination gloves (latex or nitrile)

11.30 Lens paper and lens cleaning solution

11.31 Safety glasses (Z-87 rated)

11.32 Paper towels

11.33 Kimwipes (or other appropriate wiping material)

12.0 CALIBRATION AND OPTIMIZATION OF THE PLM

12.1 Equipment and Standards

12.1.1 All measuring and testing equipment having an effect on the accuracy and/or validity of analytical testing must be calibrated at frequencies described for the individual components below.

12.1.2 "Standards" refers to any material used in calibration of a piece of equipment or analytical methodology.

12.1.2.1 Standards used at the lab include slides used for alignment of a microscope's polars, optical glass for calibration of RI liquids, NIST SRMs of the various asbestos minerals, and Controlled PE Reference Materials of LA in soils.

12.1.2.2 The laboratory uses NIST-traceable standards whenever possible, or other standards that have been calibrated by a respected organization. When internal standards are used, they are checked as extensively as technically and economically feasible.

12.1.2.3 The laboratory stores its standards in such a way to avoid contamination of the standards and to protect their integrity.

12.1.2.4 Any standard that is damaged, compromised, or judged to be unreliable must be recalled from service.

12.1.2.5 Reference standards of measurement (e.g., optical glass for RI liquid calibration, slides for aligning the microscopes, and LA reference materials) are used for calibration purposes and for no other purpose.

12.1.3 Visual estimates of asbestos concentrations other than LA, as well as LA concentrations greater than 1%, are calibrated using permanently mounted working slides of known asbestos concentration prepared by the laboratory. The use of these standards is described in Section 16.0.

12.1.4 Visual estimations of LA concentrations equal to or less than 1% are calibrated using the Controlled PE Reference Materials.

12.2 General Maintenance and Calibration of the Polarized Light Microscope

12.2.1 Chrysotile, amosite, crocidolite, and anthophyllite all have the optical property of parallel extinction. Because this is one of the optical properties used to identify these minerals, the polars of the PLM must be aligned north-south (N-S) and east-west (E-W), and the polars must be kept at 90 degrees to each other.

12.2.1.1 A mineral grain's extinction angle cannot be measured accurately if the polars are not correctly aligned.

12.2.2 LA and some non-asbestos minerals (wollastonite, hornblende, etc.) will often display an inclined (or oblique) extinction angle.

12.2.3 The lower polar must be properly aligned E-W so RI's in the parallel and perpendicular directions can be measured correctly.

12.2.4 The polars should be kept at 90 degrees to each other so the field of view in crossed polars is as dark as possible.

12.2.5 The microscope's optics must be kept clean and properly aligned so optimal image quality can be produced.

12.2.6 Check the microscope's alignment each working day prior to use.

12.2.6.1 The microscope must be re-aligned any time it is found to be out of alignment. Follow all the procedures outlined in Sections 12.3

through 12.8 for re-calibrating the microscope.

12.2.7 Each day the microscope is used, record an entry in the microscope's instrument maintenance logbook. Record the date and analyst's initials confirming that all microscope alignment checks were made prior to analysis.

12.2.7.1 An individual instrument maintenance logbook must be kept for each microscope in use at the laboratory.

12.2.7.2 All maintenance activities performed on the microscope must be recorded in the appropriate logbook.

12.2.7.3 Each day the microscope is used to analyze samples, a data entry must be made in the logbook indicating that the microscope was properly calibrated that day prior to use.

12.3 Checking Microscope Alignment

12.3.1 Place a permanently-mounted test slide that contains large straight fibers of anthophyllite or polypropylene onto the microscope stage.

12.3.1.1 While looking at an empty portion of the slide under crossed polars, make sure the field of view in the microscope is as dark as possible (black, not dark gray).

12.3.1.2 When the field of view is black under crossed polars, the polars are oriented at 90 degrees to each other.

12.3.2 The fibers of anthophyllite should be completely extinct in both the N-S and E-W directions under crossed polars, indicating proper polar alignment.

12.3.2.1 Once the fibers of anthophyllite become completely extinct in either the N-S or E-W direction, pull the analyzer out to make sure the fibers of anthophyllite are still parallel to the crosshairs.

12.3.3 The stage and objectives must be centered so that a fiber centered in the field of view remains centered in view when the microscope stage is rotated.

12.3.4 The light path through the scope must be centered (specifically, the condenser and iris diaphragm must be centered on the optic axis).

12.3.5 The crosshairs should be properly oriented E-W and N-S.

12.3.6 If any of the above conditions are not met, it is necessary to re-calibrate the microscope.

12.4 Centering the Stage and Objectives

12.4.1 Because centering of the highest magnification objective (40X or 50X) is the most critical, center the microscope stage to this objective.

12.4.1.1 Adjust the centering screws on the stage so that a particle remains centered in the field of view when using the highest magnification objective as the stage is rotated.

12.4.1.2 The remaining objective lenses must be centered so they coincide with the axis of rotation of the stage.

12.4.1.3 Adjust the centering of the remaining objectives using the centering screws for each objective.

12.5 Centering the Optic Axis

12.5.1 Looking at the field of view in plane light under low magnification, insert the sub-stage condenser lens and then tighten the field iris diaphragm (not the

condenser iris diaphragm) until it begins to eclipse the outer edge of the field of view.

12.5.2 Use the centering screws to center the image of the outer edge of the field diaphragm so it coincides with the edge of the field of view.

12.5.3 Tighten the field iris diaphragm until it is almost closed. With the 10X objective, only a small circle of light should be visible somewhere close to center of the field of view.

12.5.3.1 Raise or lower the microscope substage until the edge of the image of the field diaphragm comes into as sharp a focus as possible.

12.5.4 Move the substage with the condenser and its iris diaphragm using its adjusting screws until the small circle of light is centered in the field of view.

12.5.5 Open the field iris diaphragm until it is just barely wide enough that the entire field of view is illuminated.

12.5.6 Remove the sub-stage condenser lens.

12.6 Using the Condenser Iris Diaphragm

12.6.1 When viewing a microscope slide under plane light, adjust the iris diaphragm on the sub-stage condenser (not the field iris diaphragm) to improve contrast and the viewing of subtle shades and textures.

12.6.1.1 The iris diaphragm is not used for controlling brightness; the light source is used to control light and brightness.

12.7 Alignment of Lower Polar

12.7.1 Place the thin section containing large crystals of biotite on the microscope stage and examine it in plane light. This procedure allows for rapid and accurate alignment of the lower polar. Laboratories may use a different procedure to align the lower polar as long as it is documented in their internal SOPs.

12.7.2 Find a biotite crystal on the slide that exhibits strong cleavage traces between the sheets of mica.

12.7.2.1 The cleavage planes in the biotite crystal between the mica sheets should be as close to perpendicular with the plane of the slide as possible.

12.7.2.2 Crystals that show the strongest cleavage traces should have their cleavage plane at a high angle to the plane of the slide and will show the most distinctive pleochroism.

12.7.2.3 After selecting a biotite crystal, orient the slide so that the cleavage traces of the biotite crystal are directly E-W.

12.7.2.4 Observe the crystal's pleochroism as the stage is rotated.

12.7.2.5 While viewing the crystal in plane light, slowly rotate the lower polar clockwise or counter-clockwise until the biotite crystal is as dark as it will become.

12.7.2.6 When the cleavage traces of the biotite crystal are oriented directly E-W and the pleochroism of the crystal is as dark as possible, the lower polar is properly oriented E-W.

12.7.3 Rotate the ocular that contains the crosshair reticle until the crosshairs are oriented directly N-S and E-W.

12.8 Alignment of Upper Polar

12.8.1 Once the lower polar has been properly aligned E-W, place a permanently-mounted test slide containing large straight fibers of anthophyllite or polypropylene on the stage.

12.8.2 While looking at a portion of the slide relatively free of birefringent material, slowly rotate the upper polar until the field of view, under crossed polars, reaches maximum darkness. The field of view should be black, not dark gray.

12.8.3 Rotate the stage and observe the extinction of the anthophyllite or polypropylene fibers.

12.8.3.1 If the field of view is as dark as possible and the fibers become extinct in the N-S and E-W directions, the polars are properly aligned.

12.8.3.2 Once the fibers become completely extinct in either the N-S or E-W direction, pull the analyzer out to make sure the fibers are still parallel to the crosshairs.

12.8.3.3 If the polars are still not properly aligned, then repeat steps 12.7.1 through 12.8.3 until the microscope's polars are properly aligned.

12.9 Cleaning the Polarized Light Microscope

12.9.1 The oculars, objective lenses, and condenser should be cleaned whenever they become soiled with dust, oil, RI liquids, etc. At minimum, they shall be cleaned monthly.

12.9.2 Always use lens cleaning solution and lens paper to clean the lenses.

12.9.2.1 Do not use a dry cloth because this can scratch the surfaces of the lenses.

12.9.2.2 Avoid applying excessive pressure to the lens surface when cleaning as this could also scratch the lens.

12.9.2.3 Never use any solvents (such as alcohol, etc.) other than lens cleaning solution because this can dissolve the cement that holds the lenses together.

12.9.3 If dust gets inside the microscope, it is necessary to completely disassemble and clean the microscope.

12.9.3.1 The microscope must be re-calibrated after being re-assembled and this must be recorded in the microscope's maintenance logbook.

12.9.3.2 Disassembly of the microscope should only be performed by qualified personnel.

13.0 DETAILED METHOD FOR ASBESTOS TESTING OF SOIL AND SOIL-LIKE MATERIALS

13.1 Stereomicroscopic Examination

13.1.1 All sample preparation activities, including stereomicroscopic examination, slide mounts, etc., must be performed in a HEPA-filtered hood, class 1 biohazard hood, or glove box with continuous airflow (negative pressure).

13.1.2 Due to the sample preparation requirements described in the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, samples should never be wet. If the sample is wet, contact EPA or designate.

13.1.3 The stereomicroscope is a low magnification microscope (approximately 10X-50X) used for visual examination of specimens at a coarse scale.

Stereomicroscopic examination is especially useful for soil samples where fibers may be unevenly or thinly distributed throughout the sample.

13.1.4 Begin the analysis by pouring the entire sample out of its container onto a clean, asbestos-free substrate, such as an agate mortar, ceramic evaporating dish, watchglass, weighing dish, etc.

13.1.4.1 For fine-ground soil samples, the mass of the sample will ideally be 20 to 50 grams; however, some samples submitted to the laboratory may be larger.

13.1.5 With the stereomicroscope, visually examine the entire sample for homogeneity and the presence of any suspect fibers.

13.1.6 If individual fibers suspected of being asbestos are observed, pick out one or more of these fibers with fine forceps (or other appropriate utensil) and mount them on a glass microscope slide in an appropriate RI liquid. These sample preparations are often called "fiber-picks" and are referred to as fiber-picks in this SOP.

13.1.6.1 Each microscope slide must be wiped with lint-free wipes prior to use to avoid contamination.

13.1.6.2 Mount individual fibers in 1.550 RI oil if chrysotile is suspected, 1.620 to 1.640 RI oil if LA or anthophyllite is suspected, or 1.680 to 1.700 RI oil if amosite or crocidolite is suspected.

13.1.6.3 Only one drop of RI liquid is necessary to prepare the fiber-pick slide.

13.1.6.4 Cover this preparation with a glass cover slip and identify the fibers using PLM analysis techniques (see Section 13.5).

13.1.7 Record all stereomicroscopic findings, including sample appearance, an initial estimated percent LA, and an initial estimated percent other asbestos (chrysotile and other amphibole), in the appropriate fields on the analytical bench sheet.

13.1.7.1 Stereomicroscopic examination does not provide positive identification of asbestos fibers. Later analysis by PLM will confirm, deny, or refine the preliminary estimated percent asbestos.

13.1.7.2 The procedure for performing a calibrated visual estimate using both stereomicroscopy and PLM is described in Section 13.7.4 and Attachment 8.

13.1.8 Even if no fibers are visible, prepare the sample as described in Section 13.3.

13.2 Determination of Ashing the Sample

13.2.1 Soil samples containing a significant amount of twigs, leaves, tar, or other debris may need to be ashed prior to being prepared for random mounts for PLM.

13.2.1.1 Excessive cellulose fibers, tar or asphalt may obscure asbestos fibers, and ashing will assist in eliminating this interference.

13.2.2 Ashing consists of placing a representative portion of the whole sample into the muffle furnace to burn off organics that obscure asbestos fibers or keep the sample from breaking up on the slide during mounting. Approximately 480°C is hot enough to burn off organics without destroying the crystallinity of asbestos fibers. Do not ash the entire sample because a re-analysis of the sample may be required at a later date.

13.2.3 The ashed residue can then be examined under the stereomicroscope following the procedures in Section 13.1, above, and slide mounts can be prepared from the ashed residue for PLM analysis, according to the procedures in Section 13.3,

below.

13.2.4 Following PLM analysis, calculate the percentage of asbestos in the pre-ash sample using the equation below:

Pre-ash percent asbestos = (percent asbestos in ashed residue) * (C-A)/(B-A)

Where:

A = weight of ashing tin in grams
B = weight of sample + ashing tin in grams (pre-ash)
C = weight of sample + ashing tin in grams (post-ash)

13.2.5 Record the required gravimetric measurements and calculations listed above in Section 13.2.4 on the analytical data sheet in the comments field. Alternatively, attach a separate analytical data sheet (specific to ashing samples) with the necessary measurements, and indicate the attachment in the comments section.

13.3 Preparation of Samples for PLM Visual Area Estimation

13.3.1 Quantitative analysis preparation typically consists of preparing random mounts of a sample. The objective is to produce random mounts of a representative sub-sample from the original sample.

13.3.2 View the sample through the stereomicroscope to determine if it is sufficiently homogenized and all particles are reduced to a small enough size.

13.3.2.1 Soil samples processed according to the current revision of SOP ISSI-LIBBY-01, Soil Sample Preparation, should be ground to a maximum particle size of approximately 250 µm.

13.3.2.2 Additional homogenization of the sample at the laboratory using a mortar and pestle may be required if any remaining inhomogeneities or coarse particles are observed in the sample. When further grinding the sample, care should be taken to not pulverize the LA to a fiber size unidentifiable by PLM techniques. The material in the slide mounts must be coarse enough that fibers of LA can still be identified by PLM and still be as representative as possible of the sample as a whole.

13.3.3 Oil immersion mounts of randomly selected sub-samples of the homogenized material are prepared in RI liquids for PLM analysis.

13.3.3.1 Prepare a minimum of five random mount slides for each sample.

13.3.3.2 Each microscope slide must be wiped clean with an appropriate wipe prior to use in order to avoid contamination.

13.3.3.3 Place one to two drops of the appropriate RI liquid onto each slide.

13.3.3.3.1 Prepare at least two slides with a RI liquid in the range of 1.620 to 1.640 for easier measurement of the optical properties of LA. Generally, 1.625 RI liquid is used for LA.

13.3.3.3.2 The refractive indices of the oils used for the remaining slides is left to the analyst's discretion based upon the suspected mineralogy present in the sample material.

13.3.3.4 Use a spatula, the curved edge of a scalpel blade, or other similar utensil to collect randomly selected sub-samples of the homogenized

sample material, and place this into the RI liquid on the slides.

13.3.3.5 With the utensil, gently stir the sample material in the RI liquid to produce a homogeneous mixture.

13.3.3.6 Cover each mixture of RI liquid and sample material with a glass cover slip.

13.3.3.7 Gently agitate the mixture under the cover slip by pressing down and rubbing the top of the cover slip with something that will "grab" the cover slip and allow it to be translated from side to side, such as an etching scribe or the eraser end of a pencil.

13.3.3.7.1 Use this action to spread the mixture of RI liquid and sample material over the approximate area of the cover slip.

13.3.3.7.2 The material under the cover slip should be spread out evenly with no or very few overlapping particles.

13.3.3.8 Wipe any loose sample material or excess RI liquid from the slide with lint-free wipes.

13.3.3.9 The prepared slide can now be safely removed from the hood for analysis by PLM.

13.4 Supplemental Stereomicroscopic Evaluation

13.4.1 Following random slide mount preparation, it may be useful agitate or tap the sample container to cause the particulate to settle and the amphibole fibers to sort to the surface.

13.4.1.1 Re-examine the sample using the stereomicroscope, and repeat procedures 13.1.6, above.

13.4.1.2 This "tapping" method should only be used as a qualitative technique following random slide mount preparation, and not as a quantitative technique, because it tends to make the sample inhomogeneous.

13.4.1.3 The representative sub-sample material used for preparing random slide mounts must remain homogeneous.

13.4.2 Avoid contamination by maintaining a clean work space.

13.4.2.1 After preparing each sample, clean all work surfaces, sample substrates, utensils, and any other items that come into contact with the sample, with distilled water and paper towels.

13.4.2.2 Dispose of gloves after they become excessively dirty.

13.4.2.3 Only prepare one sample at a time. Never have more than one sample container open inside the preparation hood at any given time.

13.4.2.4 When placing drops of RI liquid on the slides, never touch the Dropper directly to a different RI oil or to oil that already has sample material in it. Only touch the dropper to a clean slide.

13.4.2.5 Discard any RI liquids that become contaminated with sample debris.

13.5 Classification of Asbestos Mineral Type

13.5.1 Analysis of Libby soil samples consists of identification and quantification of any and all asbestos phases present within the sample, and when possible, the identification and semi-quantification of non-asbestos fibers and the identification of matrix materials within the sample.

13.5.2 Positive identification of asbestos, non-asbestos fibers, and matrix material is conducted by examination of sample slide mounts by PLM.

13.5.3 Visually examine the entire area of all prepared slides using PLM (using both plane light and crossed polars) to find any fibrous constituents within the slide mounts.

13.5.4 Positive identification of asbestos requires the determination of the following six optical properties by PLM.

- 13.5.4.1 Morphology
- 13.5.4.2 Color and pleochroism (if pleochroism is present)
- 13.5.4.3 Refractive indices, both alpha and gamma
- 13.5.4.4 Birefringence
- 13.5.4.5 Extinction characteristics
- 13.5.4.6 Sign of elongation (positive if the fiber is length slow, negative if the fiber is length fast)

13.5.5 Asbestos cannot be reported in any quantity, including trace, until its optical properties have been measured and recorded.

13.5.6 Based on the optical properties, asbestos in the sample is classified into one of three categories described in Table 13.1:

Table 13.1

Code	Description	Notes
LA	Libby Amphibole	The minerals winchite, richterite, tremolite, and actinolite, which are characteristic of the mine at the Libby Superfund Site. Also included are the minerals magnesio-arfvedsonite and magnesio-riebeckite, which are known to occur at the Libby Asbestos Superfund Site in smaller quantities.
OA	Other amphibole asbestos	Regulated amphibole asbestos (amosite, crocidolite, and anthophyllite) that are not thought to occur in significant amounts at the mine in Libby.
C	Chrysotile	Asbestiform serpentine

13.5.7 Chrysotile $Mg_3Si_2O_5(OH)_4$

- 13.5.7.1 Serpentine is a phyllosilicate (sheet-silicate) mineral, and when serpentine occurs in an asbestiform morhology, it is referred to as chrysotile.
- 13.5.7.2 There are three varieties of the mineral serpentine: antigorite, lizardite, and chrysotile. All three have the same chemical composition but different morphologies.
- 13.5.7.3 Individual fibrils of chrysotile have been shown by transmission electron microscopy (TEM) to be in the form of scrolled tubes, or tightly rolled micaceous sheets, such that the fibril axis lies within the plane of the sheets (much as if a newspaper had been rolled up). In other types of serpentine, the sheets may be curved, but they are flat or platy, not rolled into tightly scrolled tubes.

13.5.7.4 If serpentine is observed and has a platy or massive (non-fibrous) morphology, it is classified as non-asbestiform serpentine (antigorite if it is platy or lizardite if it occurs as a massive, fine-grained matrix) and not as asbestos (chrysotile).

13.5.7.5 If serpentine is observed and has a fibrous morphology, it is classified as chrysotile asbestos.

13.5.7.6 The morphology of chrysotile is fibrous and sometimes silky.

13.5.7.7 The fibers are flexible. Chrysotile sometimes occurs as tangled mats of many fibers.

13.5.7.8 Chrysotile can only be seen in PLM as chrysotile bundles; the individual fibrils that make up a chrysotile bundle are beyond the resolution of all light microscopy.

13.5.7.9 Bundles of chrysotile are often splayed.

13.5.7.10 Kinked chevron-style folds are sometimes seen in chrysotile.

13.5.7.11 Chrysotile is usually colorless in PLM, although it sometimes shows a slight golden, yellow, or pale golden-green color in PLM.

13.5.7.12 Chrysotile that has been exposed to very high temperatures is distinctly brown under plain light.

13.5.7.13 Chrysotile is never pleochroic.

13.5.7.14 Small particles of opaque magnetite can sometimes be seen in large, intact bundles of chrysotile.

13.5.7.15 The range for the lower RI (alpha, or α) for chrysotile is 1.545 to 1.553 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.16 The range for the higher RI (gamma, or γ) for chrysotile is 1.552 to 1.560 as reported in the certificate for NIST SRM 1866b, although the range for chrysotile encountered in field samples may be somewhat wider.

13.5.7.17 Exposure to high heat and dehydration of the crystal lattice will increase the refractive indices of chrysotile.

13.5.7.18 The birefringence (expressed numerically as δ, the difference between α and γ) of chrysotile is low, usually around 0.008. In practice, this means that most chrysotile bundles of fine to medium size observed in samples will have low first order gray to medium gray interference colors under crossed polars. Larger, thicker fibers can show first order white to yellow interference colors; higher colors may be seen in the thickest bundles.

13.5.7.19 Chrysotile is most easily visible in plane light in the higher RI liquids, such as 1.62 or 1.68. However, measurement of the refractive indices of chrysotile should be done with the fibers mounted in the 1.550 oil.

13.5.7.20 Chrysotile is almost always length slow (positive sign of elongation), although length fast chrysotile has been observed on very rare occasions.

13.5.7.21 Chrysotile invariably has parallel extinction.

13.5.8 Amosite $Fe_7Si_8O_{22}(OH)_2$

13.5.8.1 The name amosite is derived from an acronym for "Asbestos Mines of South Africa". It is a trade name and not a mineralogical name. Amosite is the fibrous variety of the mineral grunerite.

13.5.8.2 Amosite has an acicular (needle-like) morphology. Bundles of amosite are composed of many lesser needles of amosite. Needles of amosite are often straight and only somewhat flexible.

13.5.8.3 Amosite is usually colorless, green, brown, or greenish-brown in plane light. Heated amosite is brown to dark brown and can be nearly opaque. Amosite is sometimes weakly pleochroic.

13.5.8.4 The range for the lower RI (α) for amosite is 1.675 to 1.681 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.5 The range for the higher RI (γ) for amosite is 1.697 to 1.704 as reported in the certificate for NIST SRM 1866b, although the range for amosite encountered in field samples may be somewhat wider.

13.5.8.6 Exposure to high heat and dehydration of the crystal lattice will increase the RI's of amosite.

13.5.8.7 The birefringence of amosite is moderate, usually about 0.020. Most fibers observed will have first order white to yellow interference colors under crossed polars; although, higher colors (first order magenta to second order or sometimes even higher) can be seen in the thicker bundles.

13.5.8.8 RI measurements should be done with the fibers mounted in 1.680 to 1.700 RI oil.

13.5.8.9 Amosite is length slow (positive sign of elongation).

13.5.8.10 Even though grunerite is a monoclinic mineral, the extremely fine fibers that form bundles of amosite cause amosite to have parallel extinction.

13.5.9 Crocidolite $Na_2Fe_3^{2+}Fe_2^{3+}Si_8O_{22}(OH)_2$

13.5.9.1 Crocidolite is a fairly uncommon type of asbestos.

13.5.9.2 Crocidolite has an acicular morphology very similar to that of amosite. The fibers are only somewhat flexible.

13.5.9.3 Crocidolite is distinctly blue or blue-green in plane light and is pleochroic.

13.5.9.4 Normally, the range for the lower RI (α) for crocidolite is 1.680 to 1.698 (EPA, 1993).

13.5.9.5 Normally, the range for the higher RI (γ) for crocidolite is 1.685 to 1.706 (EPA, 1993).

13.5.9.6 The strong color of crocidolite makes measurement of the refractive indices very difficult. For this reason, select finer fibers of crocidolite, which have less color, when measuring refractive indices.

13.5.9.7 The birefringence of crocidolite is low, usually about 0.006. Crocidolite often shows anomalous interference colors under crossed polars.

13.5.9.8 RI measurements on crocidolite should be done with the fibers mounted in 1.680 or 1.700 oil.

13.5.9.9 Because crocidolite is length fast, the lower RI (α) should be measured with the fiber oriented in the E-W direction (parallel to the lower polar), and the higher RI (γ) should be measured with the fiber oriented in the perpendicular (N-S) direction.

13.5.9.10 Even though riebeckite is a monoclinic mineral, the extremely narrow fibers that form bundles of crocidolite cause crocidolite to have parallel extinction.

13.5.10 Anthophyllite $(Mg,Fe)_7Si_8O_{22}(OH)_2$

13.5.10.1 Anthophyllite occurs as straight to slightly curved fibers or fiber bundles. The morphology of anthophyllite is lamellar to acicular.

13.5.10.2 Anthophyllite is a rare type of asbestos used in construction materials.

13.5.10.3 Anthophyllite is colorless to pale brown in plane light. It is sometimes weakly pleochroic.

13.5.10.4 The range for the lower RI (α) for anthophyllite is 1.593 to 1.694 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has an α of 1.615.

13.5.10.5 The range for the higher RI (γ) for anthophyllite is 1.613 to 1.722 (Deer et al., 1997). The commercial-grade anthophyllite in SRM 1867a has a γ of 1.636.

13.5.10.6 The birefringence of anthophyllite is moderate, usually about 0.020.

13.5.10.7 Generally, RI measurements on anthophyllite should be done with the fibers mounted in 1.620 to 1.640 oil.

13.5.10.8 Because anthophyllite is an orthorhombic mineral, all fibers of anthophyllite will invariably have parallel extinction. This helps to distinguish it from LA and the non-asbestos mineral wollastonite, which often show inclined extinction.

13.5.10.9 Anthophyllite is length slow (positive sign of elongation).

13.5.11 Libby Amphibole

13.5.11.1 LA consists of Tremolite-Actinolite, $Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$, Winchite, $CaNaMg_4(Al,Fe^{3+})Si_8O_{22}(OH)_2$, Richterite, $NaCaNa(Mg,Fe)_5Si_8O_{22}(OH)_2$, Magnesio-arfvedsonite, $(Na,K)Na_2Mg_4Fe^{3+}Si_8O_{22}(OH)_2$, and Magnesio-riebeckite, $Na_2Mg_3Fe^{3+}{}_2Si_8O_{22}(OH)_2$.

13.5.11.2 LA is a term used to categorize a group of minerals generally described as sodic tremolite. The solid solution series of sodic tremolite is comprised of a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below and in Attachment 4. As seen, there is a great deal of overlap in optical properties among the minerals that make up LA. As such, discreet mineral identification is not required by this SOP. Rather, if the sample exhibits the optical properties of a mineral listed in this section, the specific optical properties (such as refractive indices, birefringence, extinction angle, and sign of elongation) shall be noted on the analytical data sheet and EDD, and the mineral identified as LA.

13.5.11.3 The morphology of LA ranges from prismatic to fibrous. The fibers that form a bundle of LA may be parallel to sub-parallel, or the fibers may sometimes cross one another at various angles giving the bundle a matted appearance. The aspect ratio of the fibers is highly variable, and all tremolite, actinolite, winchite, richterite, magnesio-arfvedsonite or magnesio-riebeckite encountered in a sample should be classified as LA regardless of the aspect ratio of the individual fibers. Refer to Attachment 5 for photomicrographs that show a wide range of LA morphologies that may be encountered during PLM analysis.

13.5.11.6 Laboratories should use the Controlled Libby Amphibole Asbestos and NIST Bulk Asbestos Proficiency Testing Round M12001, Sample 4, as reference materials to familiarize themselves with the range of habits and optical properties of LA. Laboratories should contact the client or their designate if they do not have these reference materials.

13.5.11.7 Color of LA in plane light is highly varied. Tremolite is usually colorless in plane light. Actinolite is usually pale green to dark green. Darker colors and stronger pleochroism are associated with higher iron content for the tremolite-actinolite series (Deer et al., 1997). Winchite can be pale yellow, blue, blue-green, or blue-gray. Richterite can be brown, tan, pale green to dark green, pale yellow, or violet (Deer et al., 1997). Magnesio-arfvedsonite in plane light is yellowish green, brownish green, or grey-blue (Deer et al, 1997). Magnesio-riebeckite in plane light is blue, grey-blue, or pale blue to yellow (Deer et al, 1997). Winchite, richterite, magnesio-arfvedsonite, and magnesio-riebeckite can all be pleochroic.

13.5.11.8 LA generally has moderate birefringence, usually about 0.015 to 0.02.

13.5.11.9 LA usually shows inclined (or oblique) extinction, although fibers in certain crystallographic orientations will exhibit parallel extinction. The maximum extinction angle for tremolite-actinolite can be as high as 10 to 21 degrees. Winchite and richterite can show higher extinction angles, sometimes as high as approximately 30 degrees or even higher for richterite.

13.5.11.10 RI measurements on LA should be done with the fibers mounted in 1.620 to 1.640 RI oil (1.625 is a commonly-used choice).

13.5.11.11 Winchite, richterite, tremolite, and actinolite are all length slow (positive sign of elongation). Both magnesio-arfvedsonite and magnesio-riebeckite are length fast (negative sign of elongation).

13.5.11.12 On the analytical bench sheet (Attachment 1), record only one set of optical properties for LA for each sample that contains LA. Choose the fiber/and or bundle that shows the best Becke line and/or dispersion staining colors.

13.5.11.13 Refer to Attachment 4, Optical Properties of Fibrous Amphiboles, for additional information on the optical properties of LA used in LA identification.

13.6 Refractometry

13.6.1 Calibration of Refractive Index Liquids

13.6.1.1 Accurate measurement of a mineral's refractive indices begins with proper calibration of the RI liquids. Each RI liquid used for routine sample preparation and analysis must be calibrated once each month.

13.6.1.2 Prepare an oil immersion mount of the appropriate certified precision optical glass in the oil to be calibrated.

13.6.1.3 Read the laboratory's thermometer to the nearest 2° C to determine the ambient temperature t, and record the temperature on the appropriate worksheet (see page 7 of Attachment 3).

13.6.1.4 Next determine λ_0. This is the wavelength at which the RI of the oil is equal to the RI of the certified precision optical glass. Observe the

central stop dispersion staining color shown by the glass, and consult the dispersion staining color chart (McCrone, 1987). If the glass particles show a range of dispersion staining colors, use the most predominant color when determining λ_0. Record the predominant dispersion staining color and corresponding λ_0 on the worksheet.

13.6.1.5 Consult the Excel spreadsheet developed by Shu-Chun Su, Ph.D., "Create_RI_Liquid_Calibration_Conversion_Tables.xls", for the appropriate conversion table (see Attachment 2). These tables are used to convert λ_0 and t into n_d^{25}, which is the calibrated RI of the oil at a wavelength of 589 nm and a temperature of 25°C. Determine the value of n_d^{25} from the appropriate table for the known values λ_0 and t.

13.6.1.6 Additional conversion tables for oils not included in the spreadsheet can be generated by entering the dispersion coefficients and values of n_d of the oil and the glass, and the value of dn/dt (change of RI with temperature) of the oil into the first sheet of the workbook. All of these values are clearly provided by the manufacturer of the glass and oil.

13.6.1.7 Record the value of n_d^{25} on the worksheet. This is the calibrated RI of the oil at a standard temperature of 25°C.

13.6.1.8 Write this calibrated RI and the date of calibration on the bottle.

13.6.1.9 If the difference between the actual calibrated RI of the oil and the original RI of the oil is greater than 0.004, then the oil may not be used for analysis of samples.

13.6.1.10 Repeat the above steps for each oil in routine use.

13.6.2 Measurement of refractive indices (refractometry) of minerals is performed using either the Dispersion Staining Method or the Becke Line Method.

13.6.2.1 All analysts must be proficient in both methods. The choice of which method to use is left to the analyst's discretion.

13.6.2.2 The dispersion staining method requires a clean surface of the mineral to be in direct contact with the oil and can only be performed if a conversion chart has been developed beforehand for a specific mineral in a specific RI liquid.

13.6.2.3 The Becke Line Method will often work on relatively fine fibers, and also requires a clean surface of the mineral to be in contact with the oil. However, this method does not require a specific mineral-oil chart to be developed before it is used. For this reason the Becke Line method can be used to measure the RI's of other materials besides LA and regulated asbestos minerals.

13.6.3 Measurement of Refractive Indices by the Dispersion Staining Method

13.6.3.1 Mount the fibers in the appropriate oil (1.550 for fibers suspected of being chrysotile, 1.620 to 1.640 oil for fibers suspected of being LA or anthophyllite, or 1.680 to 1.700 oil for fibers suspected of being amosite or crocidolite).

13.6.3.2 In order for the correct dispersion staining colors to be displayed, a clean surface of the mineral must be in direct contact with the RI liquid.

13.6.3.3 If may be necessary to separate and spread out fibers bundles on the slide so a clean surface is exposed. Do this by agitating the bundles with an X-acto knife or other sample manipulation utensil, or rubbing the cover slip over the bundles to agitate and dis-aggregate them.

13.6.3.4 Examine the slide in plane light using the 10X dispersion staining objective. The dispersion staining objective and its central stop should be centered.

13.6.3.5 Stop down the condenser iris diaphragm until dispersion colors are observed.

13.6.3.6 Read the thermometer to find ambient temperature of the laboratory's air to the nearest 2°C.

13.6.3.7 To measure α, orient the fiber E-W (parallel to the lower polar) if the fiber is suspected of being crocidolite, or N-S if the fiber is suspected of being chrysotile, amosite, or anthophyllite. LA shows biaxial optics and requires a more detailed treatment, described below in Section 13.6.5.

13.6.3.8 Next, observe the dispersion staining color that is displayed.

13.6.3.9 Light of a wavelength higher or lower than the matching wavelength (given the symbol λ_0, where the RI of the oil matches the RI of the mineral) is refracted around the central stop and passes through.

13.6.3.10 Light of a wavelength equal or approximately equal to the matching wavelength is blocked.

13.6.3.11 The observed color is the summation of the remaining light.

13.6.3.12 Consult the dispersion staining color chart (McCrone, 1987) and find the matching wavelength (λ_0) that corresponds to the observed color.

13.6.3.13 When measuring α and a range of dispersion staining colors is displayed, choose the color that produces the lowest RI, i.e., the color that corresponds to the longest λ_0.

13.6.3.14 Refer to the paper "Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers by Using Dispersion Staining Method", by Shu-Chun Su, Ph.D. (1996).

13.6.3.15 For the appropriate RI oil and mineral combination, find the column for the laboratory's temperature and row for λ_0; record the corresponding value of RI.

13.6.3.16 To measure γ, rotate the stage 90 degrees.

13.6.3.17 The fiber should now be perpendicular to the lower polar (N-S) if the fiber is suspected of being crocidolite, or parallel to the lower polar (E-W) if the fiber is suspected of being chrysotile, amosite, or anthophyllite. Refer to Section 13.6.5 for orienting fibers of LA when measuring γ.

13.6.3.18 Observe the dispersion staining colors and find the corresponding λ_0. When measuring γ, choose the color that produces the highest RI, i.e., the color that corresponds to the shortest λ_0.

13.6.3.19 Consult the appropriate chart for the asbestos type and oil being used; record the value of RI for the temperature and λ_0.

Note: There are two charts for each mineral and oil combination - one for α and one for γ. Be sure to use the appropriate chart when measuring α or γ.

13.6.4 Measurement of Refractive Indices by the Becke Line Method

13.6.4.1 Becke line colors are observed in plane light when the RI of the mineral is close to or the same as the RI of the oil. Becke line colors are usually best observed using high magnification (200X to 500X).

13.6.4.2 To measure refractive indices using the Becke line method, mount the fibers in an oil whose RI is close to that of the mineral.

13.6.4.3 Observe the Becke line colors with the fiber oriented in the parallel and perpendicular directions.

13.6.4.4 As a rule, the Becke line moves into whichever medium (the grain or the oil) that has a higher RI when the microscope stage is lowered from the focused position.

13.6.4.5 Colored Becke lines are produced when the RI of the grain is higher than the oil for some wavelengths of light in the visible spectrum and when the RI of the grain is less than the oil for other wavelengths.

13.6.4.6 If a brownish or rust colored Becke line moves into the grain when the microscope stage is lowered, and a bluish-white Becke line moves into the oil, the RI of the grain is less than that of the oil.

13.6.4.7 If an orange-yellow, yellow, or lemon-yellow Becke line moves into the grain when the stage is lowered, and a violet or blue-violet Becke line moves into the oil, the RI of the grain is higher than that of the oil.

13.6.4.8 A perfect match occurs when n_d (the RI for the wavelength of sodium light, 589 nm) is the same for both the grain and the oil. When the n_d of mineral matches the n_d of the oil, an orange Becke line with just a touch of red moves into the grain and a bluish line moves into the oil when the stage is lowered.

13.6.4.9 If a perfect match cannot be obtained, mount the mineral in two oils that bracket the RI of the mineral, and interpolate where the RI of the mineral should be.

13.6.4.10 The Becke Line Chart by F. D. Bloss (1999) may be used to approximate the size of the difference between the RI of the oil and the RI of the mineral.

13.6.5 Biaxial Optics

13.6.5.1 Anthophyllite and LA often show biaxial optics. This is rarely a consideration for amosite or crocidolite.

13.6.5.2 Even though chrysotile is a monoclinic mineral, it does not show biaxial optics because of the scrolled nature of the fibers.

13.6.5.3 When an asbestos fiber shows biaxial optics, it is easy to measure a RI called α' that is between true α and beta (β) when attempting to measure α.

13.6.5.4 True α can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.5 For the monoclinic minerals that display biaxial optics (LA), the crystals need to be oriented so the X and Z axes of the biaxial indicratix corresponding to the directions of α and γ are parallel to the lower polar when measuring these indices, and they are not necessarily oriented with the crystallographic axes. As a general rule, when these fibers show inclined extinction, select the fibers that show the highest extinction angle when measuring α and γ. RI measurements should be made on a fiber where the plane of X and Z in the biaxial indicatrix lies as close to parallel to the plane of the microscope stage as possible, such that the microscopist is looking directly down Y, which corresponds to the β RI (and also the b crystallographic axis for tremolite, actinolite, winchite, richterite, and magnesio-arfvedsonite). Fibers at or close to this orientation will tend

to show the highest extinction angle.

13.6.5.6 Next, when measuring α for LA, orient the fiber approximately N-S, at the orientation where the fiber is extinct under crossed polars. The fiber should now be oriented away from N-S at an angle that is equal to its extinction angle.

13.6.5.7 Repeat this for a number of crystals. If the crystals show different Becke line colors or dispersion staining colors, measure α for the crystals that display the lowest RI.

13.6.5.8 Similarly, it is easy to measure a RI called γ' that is between β and true γ when attempting to measure γ. True γ can only be observed when a crystal is oriented in exactly the correct position.

13.6.5.9 Orient a fiber of LA approximately E-W, so that the fiber is extinct under crossed polars, when measuring γ. The fiber should now be oriented away from E-W at an angle equal to its extinction angle, so that the Z direction of the biaxial indicatrix is parallel to the lower polar. Repeat this for a number of crystals. If the crystals show different Becke line colors or dispersion staining colors, measure γ for those that display the highest RI.

13.6.5.10 Biaxial Optics of Anthophyllite

13.6.5.10.1 When measuring α (the lower RI) for anthophyllite, the fiber should be oriented in the perpendicular (N-S) direction. When fibers of anthophyllite are oriented in the N-S position, they can show either α or β, or anywhere in between, depending on their orientation. It is therefore necessary to examine a number of fibers oriented in the N-S position to find true α (α will be shown for the fibers that show the lowest RI).

13.6.5.10.2 When measuring γ (the higher RI) for anthophyllite, the fiber should be oriented in the parallel (E-W) position. Fibers of anthophyllite lying flat on the slide will always show γ and not γ', because the c-axis of the fiber will lie within the plane of the slide.

13.7 Quantification of Asbestos Content

13.7.1 General

13.7.1.1 Asbestos is reported as mass fraction percent for LA and is reported as area fraction percent for chrysotile, amosite, crocidolite, and anthophyllite.

13.7.1.2 Asbestos must be positively identified, and its optical properties measured and recorded, before asbestos can be reported in any quantity, including trace.

13.7.1.3 Quantification of asbestos concentration is performed by making a calibrated visual estimate by PLM on carefully prepared slide mounts of the sample material, in conjunction with stereomicroscopic examination of the sample.

13.7.2 Calibrated Visual Estimate of Asbestos Concentration by PLM

13.7.2.1 To perform a calibrated visual estimate, first decide on the best optical set-up to maximize the contrast between asbestos and non-

asbestos phases within the slide mounts.

13.7.2.2 Higher magnifications (200X or 400X) will improve the visibility of asbestos when it is very fine. Lower magnification (100X) should be used when the asbestos is coarse. Use of the compensator plate under crossed polars enhances the contrast between asbestos and non-asbestos on some samples.

13.7.2.3 Scan the entire area of the slides, paying attention to the relative proportion of asbestos to non-asbestos.

13.7.2.4 Draw on previous experience to make a precise calibrated visual estimate. Making accurate calibrated visual estimates is a skill that must be learned and analysts generally improve over time.

13.7.3 Use of Reference Materials for Visual Estimation of Asbestos Content

13.7.3.1 Visual area estimation is a semi-quantitative approach requiring the microscopist to estimate the area fraction of asbestos as a percentage of the total material present over many fields of view. Area fraction estimation may be difficult, especially at low concentration values and because the desired output for LA is an estimate of mass fraction (percent asbestos by weight). As a result, all visual estimates of LA content will be performed using a set of site-specific reference materials (calibration standards) as a frame of reference. These Controlled PE Reference Materials will contain either 0.2% or 1.0% LA by weight[1] and were prepared for analysis using the same approach as for field samples.

13.7.3.2 Labs analyzing samples for LA should prepare five slide mounts each of the 0.2% and 1.0% Controlled PE Reference Materials in a permanent medium, such as epoxy or melt-mount. These permanently-mounted slides can then be readily referred to by analysts as needed. When using the 0.2% and 1.0% standards as calibration materials for visual estimates, always examine the entire area of all five slide preparations by PLM for each of these standards. This will guard against potential analytical bias that may be introduced by inhomogeneities in the calibration standards.

13.7.3.3 Photomicrographs of representative fields of view of the 0.2% and 1.0% LA reference materials are included as Attachment 7 of this SOP so that analysts may refer to them as needed.

13.7.3.4 Note that because these reference materials are based on LA, they are not appropriate for estimating the mass percent of other types of asbestos (chrysotile, amosite, crocidolite, or anthophyllite). Therefore, if any asbestos types besides LA are observed, the reported values for those asbestos types should be in units of area percent.

[1] The nominal mass fraction of the reference materials (calibration standards) is based on the gravimetric fraction of the material that is soil and the amount that is spiking material, adjusted for the fraction of the spiking material that is LA. For example, if the spiking material were estimated to contain 85% LA by mass, then the 1.0% calibration standard would contain 1.18 grams of spiking material (1.00 grams of LA) per 100 grams of calibration standard. Because the estimate of LA content of the spiking material is approximate, the true concentration of a calibration material may not be precisely equal to the nominal value.

13.7.3.5 It is recommended that laboratories prepare their own permanently-mounted slides of other asbestos types (such as amosite and chrysotile) in low concentrations. This can be performed by weighing out small quantities of relatively pure asbestos (such as NIST SRM's 1866b and 1867a) and a non-asbestos matrix material (such as calcite or gypsum). The two fractions can then be mixed together, and the mixture can be mounted on a slide in a permanent medium, such as epoxy or melt-mount.

13.7.3.6 Visual comparison charts can be posted on the walls of the PLM laboratory within sight of the microscope(s) so that analysts may refer to them as necessary. A number of these charts are available, such as the Comparison Chart for Visual Percentage Estimation (after Terry and Chilingar, 1955) and the visual estimation charts developed by Dr. Shu-Chun Su (see References).

13.7.3.7 For LA, compare what is seen in the 0.2% and 1.0% Controlled PE Reference Materials and visual comparison charts as needed. The concentrations of LA in the 0.2% and 1.0% reference materials were placed at the "bin cut-offs" that place LA concentrations of each sample into one of four categories (see Section 13.8.5, below).

13.7.3.8 Other LA reference materials, such as the 0.5% and 2.0% reference materials, may also be used for comparison when performing visual estimates. However, analysts should rely primarily on the 0.2% and 1.0% Controlled PE Reference Materials for assignment of samples to bin categories; the other reference materials should be used only as supporting tools for determining LA content.

13.7.4 Combining Stereomicroscopic and PLM Visual Estimates

13.7.4.1 Analysts must not place over-reliance on either stereomicroscopy or PLM when performing visual estimates. The advantage of stereomicroscopy is that the entire sample can be examined. However, once fibers are smaller than a certain size (approximately 250 µm or less in length) it becomes difficult to impossible to find them with the stereomicroscope and mount them in a RI liquid for positive identification by PLM. Conversely, only a small sub-sample of the whole sample is examined in the random slide mounts prepared for PLM analysis. This means a PLM result can be biased high or low if the prepared slides are not representative of the sample as a whole. Therefore, it is necessary to base a calibrated visual estimate of asbestos content on both detailed stereomicroscopic observation of the entire sample and examination of the entire area of all five prepared slide mounts by PLM, as both microscopic tools are complementary to one another.

13.7.4.2 Examine every sample stereomicroscopically to produce an initial estimate of asbestos content. As described in Section 13.2 of this SOP, this preliminary stereomicroscopic visual estimate of asbestos content is recorded on the analytical bench sheet.

13.7.4.3 Carefully analyze the entire area of all five prepared slide mounts of the sample by PLM. The PLM result is then compared to the original stereomicroscopic estimate of asbestos concentration. The PLM result will confirm, refine, or deny the original stereomicroscopic estimate.

13.7.4.4 The PLM result may indicate the need to re-examine the sample stereomicroscopically, and possibly, the need to re-mount and re-analyze the sample by PLM.

13.7.4.5 Decide what asbestos concentration to report based on both the stereomicroscopic estimation of asbestos content and the PLM visual estimate of asbestos content. Stereomicroscopic examination is often an iterative process used in conjunction with analysis by PLM. Refer to Attachment 8 for a flow diagram describing this entire process.

13.7.4.6 If the asbestos is fine, more weight should be placed on the PLM mounts when estimating asbestos content. If the asbestos is coarse, more weight should be placed on the stereomicroscopic estimate. However, both stereomicroscopic examination and PLM are required for every Libby soil sample analyzed at the laboratory.

13.7.4.7 If different asbestos concentrations are observed in the different slide mounts, then the PLM estimate should be an average of all prepared slides.

13.7.5 LA Bin Categories

13.7.5.1 All winchite, richterite, tremolite, actinolite, magnesio-arfvedsonite, and magnesio-riebeckite observed in a sample is counted as LA and contributes to the bin category (described in Table 13.2), regardless of its morphology type or aspect ratio. This includes prismatic LA, as well as more fibrous varieties, such as bundles with fibers crossing at various angles giving the bundle a "matted" appearance. Refer to Attachment 5 for examples of a wide range of LA morphologies. Also refer to Attachment 6 for photomicrographs of representative examples of LA morphologies as imaged by the United States Geological Survey (USGS) by Scanning Electron Microscopy (SEM).

13.7.5.2 Using the two Controlled PE Reference Materials (0.2% and 1.0%) as a visual guide, the microscopist will evaluate the sample and report LA results as follows:

Table 13.2

PLM Laboratory Report			Description
Qual	Conc (wt.%)	Bin	
ND		A	LA was not observed in the sample
Tr		B1	LA was observed in the sample at a level that appeared to be lower than the 0.2% reference material
<	1	B2	LA was observed in the sample at a level that appeared to be approximately equal to or greater than the 0.2% reference material but was less than the 1% reference material.
	1, 2, 3, etc	C	LA was observed in the sample at a level that appeared to equal or exceed the 1% reference material. In this case, the mass percent is estimated quantitatively.

13.7.5.3 **"ND" (not detected) in the Qualifier column** is used for all samples in which LA is not observed using stereomicroscopy and is also not detected in each of a minimum of five different PLM slides prepared using representative sub-samples of the test material. These samples are assigned to **Bin A**.

13.7.5.4 **"Tr" (trace) in the Qualifier column** is used for all samples in which LA is observed either using stereomicroscopy or in at least one of the five required PLM slides prepared from representative sub-samples of the test material, and in which the amount of LA present appears to be less than the 0.2% reference material. These samples are assigned to **Bin B1**.

13.7.5.5 **"<" (less than) in the Qualifier column and "1" in the Concentration column** is used for all samples in which LA is observed either by stereomicroscopy or by PLM in slides prepared from representative sub-samples of the test material, and in which the average amount of LA present appears to be equal to or greater than the 0.2% reference material but less than the 1% reference material. These samples are assigned to **Bin B2**.

13.7.5.6 **A numeric value (1, 2, 3, etc.) in the Concentration column without an entry in the Qualifier column** is used for all samples in which LA is observed either by stereomicroscopy or by PLM in slides prepared from representative sub-samples of the test material, and in which the average amount of LA present appears to be equal to or greater than the 1% reference material. These samples are assigned to **Bin C**.

13.7.6 Visual Estimations for Chrysotile, Amosite, Crocidolite, and Anthophyllite

13.7.6.1 Visual estimates for chrysotile, amosite, crocidolite, and anthophyllite are reported as area percent.

13.7.6.2 Do not use the bins designed for LA content for concentrations of chrysotile, amosite, crocidolite, and anthophyllite. Rather, report area fraction as ND if these analytes are not detected, "<1" if these analytes were detected but at a concentration of less than 1% by area, or to the nearest whole percentage (1%, 2%, 3%, etc.) if these analytes were detected at a concentration of 1% or higher.

13.8 Non-Asbestos Fibrous Constituents

13.8.1 When non-asbestos fibers are observed, measure and record on the bench sheet at least one optical property that distinguishes the fiber from asbestos.

13.8.2 There are several non-asbestos fibers that can be confused with asbestos, and the analyst must be aware of their properties and morphologies. Commonly encountered non-asbestos fibers are listed below.

13.8.3 Talc $Mg_3Si_4O_{10}(OH)_2$

13.8.3.1 Talc usually occurs in a platy or fibrous morphology that looks similar to that of chrysotile.

13.8.3.2 Talc has a higher birefringence than chrysotile.

13.8.3.3 The birefringence of talc is in the range of 0.03 to 0.05 which gives relatively fine fibers of talc first order white to yellow interference

colors under crossed polars. Chrysotile fibers of comparable size would have low first order gray interference colors.

13.8.3.4 Talc has higher refractive indices (α = 1.54 to 1.56, γ = 1.57 to 1.60) than chrysotile.

13.8.3.5 Talc's refractive indices are less than those of tremolite, actinolite, or anthophyllite.

13.8.4 Wollastonite $CaSiO_3$

13.8.4.1 Wollastonite is one of the pyroxenoid minerals and has a characteristically bladed or prismatic morphology.

13.8.4.2 Wollastonite is colorless in plane light.

13.8.4.3 The refractive indices of wollastonite are very close to that of tremolite. However, wollastonite has a lower birefringence (difference between α and γ = 0.013 to 0.017) than tremolite.

13.8.4.4 Wollastonite has an extinction angle of up to approximately five degrees, which makes it easy to confuse with tremolite.

13.8.4.5 Crystals of wollastonite can be spun about their long axis until they change from length slow to length fast or vice versa. Crystals of tremolite will always remain consistently length slow regardless of their optical orientation.

13.8.4.6 One way to spin a wollastonite grain about its long axis is to agitate the mixture of RI liquid and sample material by repeatedly tapping the cover slip with the point of a ball point pen. Unless the crystals are lying flat on one crystal face, they should rotate as the RI liquid is agitated.

13.8.5 Kyanite Al_2SiO_5

13.8.5.1 Kyanite is an orthosilicate mineral that is commonly used in refractory materials.

13.8.5.2 Kyanite usually has a bladed or columnar morphology.

13.8.5.3 Kyanite is colorless to light blue in plane light. Its blue color is much more subdued than that of crocidolite.

13.8.5.4 Kyanite has positive relief in 1.680 oil. Its refractive indices are higher than those of crocidolite or amosite (for kyanite, α = 1.710 to 1.718, γ = 1.724 to 1.734).

13.8.6 Hornblende $(Ca,Na)_{2\text{-}3}(Mg,Fe,Al)_5Si_6(Si,Al)_2O_{22}(OH)_2$

13.8.6.1 Hornblende is one of the most common amphiboles, often found in soils in areas where certain types of igneous and metamorphic rocks are found. Hornblende is often found in soil samples from the Libby area.

13.8.6.2 Edenite, $NaCa_2(Mg,Fe^{2+})_5Si_7AlO_{22}(OH)_2$, is an amphibole that may be present at the mine at Libby (Meeker et al. 2003). Edenite is part of the hornblende group, and for this reason, for the purposes of this SOP, should not be classified as LA if it is encountered in a field sample.

13.8.6.3 Hornblende generally has slender prismatic to bladed crystals. The traces of cleavage planes are usually visible within of the crystals.

13.8.6.4 Hornblende does not occur in a highly fibrous morphology like LA often does.

13.8.6.5 Hornblende is distinctly colored and pleochroic. Hornblende is usually green, yellow-green, brown, green-brown, or blue-green in plane light.

13.8.6.6 Refractive indices vary with composition, but usually α = 1.645 to 1.665 and γ = 1.660 to 1.690. This is higher than LA.

13.8.6.7 Birefringence is moderate.

13.8.6.8 Hornblende can have parallel or inclined extinction depending on optical orientation. When extinction is inclined, the extinction angle is usually 14 to 25 degrees.

13.8.7 Calcic Clinopyroxene

13.8.7.1 The calcic clinopyroxene group includes Augite, $(Ca,Na)(Mg,Fe,Al)(Si,Al)_2O_6$, and the end members Diopside, $CaMgSi_2O_6$, and Hedenbergite, $CaFeSi_2O_6$. These are mentioned here because they are among the most common pyroxenes, but analysts should be aware that there are others.

13.8.7.2 Calcic clinopyroxene can be found in soils from areas where certain types of igneous and metamorphic rocks occur and has been found in field samples from the Libby area.

13.8.7.3 The morphology of calcic clinopyroxene is usually prismatic to columnar. As a group, the pyroxenes tend to form less slender, elongated crystals than the amphiboles. Traces of cleavage planes are usually visible within crystals of the pyroxenes.

13.8.7.4 Augite is colorless, pale green, greenish-brown, pale brown, or gray in plane light. Diopside is colorless, but as iron content increases through the diopside-hedenbergite, the mineral develops a green color.

13.8.7.5 Calcic clinopyroxene can be weakly pleochroic.

13.8.7.6 Calcic clinopyroxene has high refractive indices (α = 1.66 to 1.75, γ = 1.69 to 1.77). The pyroxenes as a group tend to have high refractive indices.

13.8.7.7 Birefringence is moderate, as with the majority of other pyroxenes.

13.8.7.8 Calcic clinopyroxene can have a very high extinction angle, up to 48 degrees.

13.8.7.9 Calcic clinopyroxene is generally length slow, but the sign of elongation becomes ambiguous in crystals showing a very high extinction angle.

13.8.8 Fiberglass (Amorphous Silica, SiO_2)

13.8.8.1 Fiberglass is almost always isotropic (appears black at all orientations under crossed polars).

13.8.8.2 Some fiberglass that is coated with other materials, or fiberglass that has been de-vitrified (partial re-crystallization of amorphous silica) due to prolonged exposure to very high temperatures, may show some slight interference colors under crossed polars.

13.8.8.3 The morphology of fiberglass is usually straight, solid, cylindrical tubes. Usually the diameter of the tube varies little along the length of the fiber.

13.8.8.4 Most fiberglass is colorless under plane light. However, the addition of impurities can impart various colors to fiberglass. Some can be yellow, dark brown, or dark green.

13.8.8.5 The RI of fiberglass varies considerably depending on the glass's composition (i.e. the addition of impurities, such as aluminum or iron). However, the RI of most types of fiberglass is close to 1.6.

13.8.9 Cellulose

13.8.9.1 Cellulose often has the morphology of ribbons that are wider than they are thick. The interiors of cellulose fibers often show a cellular or structured network.

13.8.9.2 Cellulose can be straight, curved, kinked, or crooked.

13.8.9.3 Cellulose is usually colorless under plane light, although it can be yellow, tan, or brown. Sometimes it has been dyed to various colors, such as red, blue, green, etc.

13.8.9.4 Cellulose displays undulatory (incomplete) extinction.

13.8.9.5 Cellulose usually has a higher birefringence than chrysotile.

13.8.9.6 Fibers of cellulose will often show first order white or yellow or higher interference colors under crossed polars.

13.8.10 Diatoms

13.8.10.1 Diatoms are minute organisms that live in both salt and freshwater and secrete shells of amorphous silica. When they die, their shells accumulate to form what is called diatomaceous earth. This diatomaceous earth is mined and is used in a variety of construction materials.

13.8.10.2 Not all diatoms are fibrous, but many are.

13.8.10.3 Fibrous diatoms generally have the morphology of cylindrical tubes, sometimes with tapered ends.

13.8.10.4 When fibrous diatoms are found in a sample, other diatoms having circular or other various (elliptical, lenticular, etc.) shapes are often found in the same sample.

13.8.10.5 Many diatom shells have complex internal structure.

13.8.10.6 Because they are made of amorphous silica, diatoms as a rule are isotropic. However, extreme heating or diagenetic processes can lead to de-vitrification, causing some diatoms to become weakly birefringent as a result.

13.8.11 Hair

13.8.11.1 Hair is usually cylindrical in shape; many fibers of hair are tapered.

13.8.11.2 Hair is usually colorless, tan, brown, or red-brown in plane light.

13.8.11.3 A central canal is often visible in hair fibers.

13.8.12 Synthetic Fibers

13.8.12.1 Synthetic fibers can be any color, including clear, pink, red, purple, blue, green, yellow, etc.

13.8.12.2 Synthetic fibers typically lack the splayed ends that chrysotile bundles commonly exhibit. Many synthetic fibers display a cylindrical morphology.

13.8.12.3 Synthetic fibers almost always have high to very high birefringence (0.1 or higher).

13.8.12.4 Many synthetic fibers show parallel extinction.

13.8.12.5 The synthetic fiber polyethylene has a wispy habit very similar to that of chrysotile.

13.8.12.6 Polyethylene has a higher birefringence than chrysotile.

13.8.12.7 Polyethylene fibers will melt if the slide is placed on the hot plate whereas chrysotile will not.

13.8.13 Rutile (TiO_2)

13.8.13.1 Titanium oxide occurs naturally as the mineral rutile, TiO_2. Rutile generally occurs as small prisms or fine acicular needles.

13.8.13.2 Refractive indices are extremely high (α = 2.6 to 2.7, γ = 2.8 to 2.9).
13.8.13.3 Rutile can be gray, brown, reddish-brown, or nearly opaque.
13.8.13.4 Needles of rutile have high birefringence, are length slow, and show parallel extinction.
13.8.13.5 Rutile occurs as an accessory mineral in certain types of igneous rocks, and because of its durability and resistance to weathering, it can sometimes be found as very small loose needles in soils. Rutile can sometimes be seen as needles that are inclusions in quartz crystals and are referred to as rutilated quartz.

14.0 RECORDING DATA AND RESULTS

14.1 Data Recording Sheets

14.1.1 Analysts record, by hand, on analytical data recording sheets (bench sheets), analytical results at the time the observations are made. Refer to Attachment 1 for a PLM-VE data recording sheet.
14.1.1.1 Additional bench sheets may be created by the laboratory as long as all of the required fields are included.
14.1.2 Completed bench sheets are the original, hard-copy records on which test data on client samples is stored.

14.2 Stereomicroscopic Examination Reportables

14.2.1 Homogeneity (Yes or No)
14.2.2 Sample appearance, including color and texture
14.2.3 Estimated percent LA
14.2.4 Estimated percent other asbestos (other amphibole and chrysotile)

14.3 Reporting Positive Asbestos Results

14.3.1 If asbestos is positively identified in the sample, record the following data for each asbestos type that is present in the sample.
14.3.2 Morphology
14.3.3 Fiber color
14.3.4 Pleochroism (Yes or No)
14.3.5 Indices of refraction (α and γ)
14.3.6 Birefringence
14.3.6.1 Low if birefringence is less than 0.010; medium if birefringence is 0.010 to 0.050; high if birefringence is greater than 0.050.
14.3.7 Extinction characteristics
14.3.7.1 Parallel or oblique/inclined
14.3.8 Sign of elongation (positive or negative)
14.3.9 Qualifier and percentages of the following materials in the sample
14.3.9.1 LA
14.3.9.2 Other amphibole (amosite, anthophyllite, or crocidolite)
14.3.9.3 Chrysotile
14.3.10 Bin assignment for LA (see Section 13.7.5)

14.4 Other Reportables

14.4.1 Record the percent non-asbestos fibrous materials, such as fibrous glass, cellulose, synthetic fibers, etc.
14.4.1.1 Record at least one optical property that identifies the material as a non-asbestos fiber (see Section 13.8).
14.4.2 Record the identity of the matrix material(s), if known.
14.4.3 Record if there was any deviation from the SOP or the analytical method.
14.4.4 Record the QA type as Not QA, Laboratory Duplicate – Self-check (LDS), or Laboratory Duplicate – Cross-check (LDC).
14.4.5 Record any pertinent comments.
14.4.6 Sign or initial the bench sheet, and record the date of analysis.

15.0 DATA REPORTING

15.1 EDD Report Generation

15.1.1 Results of PLM analyses are provided to the client in an EDD.
15.1.2 All of the data recorded on the bench sheet is entered into an EDD in the form of an Excel spreadsheet.
15.1.2.1 The EDD was developed specifically for the Libby project, and the laboratory should check with the client to be sure it is using the most recent version of the spreadsheet.
15.1.2.2 Only one EDD is produced for each work order number.
15.1.2.3 Data entry instructions are provided on the spreadsheet.
15.1.3 After entering all data into the EDD, save the file by clicking on the macro button located on "Visual_data entry" worksheet.
15.1.3.1 The file name is generated automatically by concatenating Information entered on the "General_data entry" worksheet.
15.1.3.2 The information used to create the file name is the laboratory name, work order number, and analysis type (visual estimation).
15.1.4 The directory where the macro will save the file depends on how the template spreadsheet was opened.
15.1.4.1 Be sure there is a blank spreadsheet template in each folder where EDD's will be saved.
15.1.4.2 If Excel is opened, and then the blank template spreadsheet is opened, the file will be saved in the same directory where the original blank template spreadsheet was opened from.
15.1.4.3 Do not open the blank template spreadsheet from Windows Explorer, because then the file will be saved at the computer's default directory for Excel (generally, this default directory is C:\Documents and Settings\My Documents).
15.1.5 The EDD serves as an electronic version of the test report submitted to the client.
15.1.5.1 A hard copy of the test report is also mailed or couriered to the client following delivery of the EDD (see Section 15.3 for further details about hardcopy data reports).
15.1.5.2 The laboratory retains all original records for use in resolving any questions until otherwise instructed by EPA.

15.2 Data Package Generation

15.2.1 Hardcopy reports of the raw analytical data are submitted to EPA, or their project oversight contractors, for archival.

15.2.2 A completed data package consists of a cover sheet signed and initialed by approved signatories and the following documentation:

15.2.2.1 Number of samples received, and copies of the signed chains of custody.

15.2.2.2 The date of sample receipt and condition of samples.

15.2.2.3 The Case Narrative, including any opinions and interpretations; deviations, modifications, additions to, or exclusions from the test method; descriptions of any problems encountered in the analysis; or any specific conditions that could affect the results. Also include the following disclaimer: "This test report relates only to items tested."

15.2.2.4 Verification that microscope slides were wiped clean before use.

15.2.2.5 Calibration data for the RI liquids used in the analysis.

15.2.2.6 Verification that the microscope was properly calibrated before use.

15.2.2.7 Verification that reference materials were used for comparison when performing calibrated visual estimates of asbestos content.

15.2.2.8 Visual Estimate hard copy data forms, as presented in the EDD and containing the analytical data (including all cross-check and self-check QC's performed on any samples in the work order number).

15.2.2.9 Copies of the handwritten bench sheets containing the analyst's original data and observations.

15.2.3 Refer to Attachment 3, the Data Package Checklist, for a complete list of items required for each data package.

15.2.4 Each test report is identified by a unique Laboratory Information Management System (LIMS) number called a Work Order Number, Job Number, or equivalent.

15.2.5 When opinions and interpretations are provided in a test report, the laboratory will:

15.2.5.1 Document the basis on which the opinions and interpretations were made.

15.2.5.2 Clearly indicate on the test report which items are opinions and interpretations.

15.2.6 Once the data package is complete, all pages must be paginated prior to delivery to the client.

15.3 Delivery of Results to Client

15.3.1 The following items will be submitted electronically (via e-mail) to the client:

15.3.1.1 The completed EDD containing the analytical data. This spreadsheet is presented in a format that can be imported into the EPA's data management software.

15.3.1.2 A scanned .pdf of all items in the data package described above, including the cover sheet signed by an approved signatory, the signed chains of custody, and the analyst's original bench sheets. All signatures must be originals, or if electronic signatures are used, the e-signature must be controlled by a password-protected login that allows its application only by the signer.

15.3.1.3 The two above files are e-mailed to the client, including all parties on

the distribution list submitted by the client to the laboratory.

15.3.2 Once the results of a work order number have been delivered to the client, a hardcopy of the data package is sent to the client through the mail.

16.0 QUALITY ASSURANCE AND QUALITY CONTROL

16.1 General

16.1.1 The laboratory operates under a quality system appropriate to the type, range, and volume of testing work that it performs.

16.1.2 Results of QC analyses are used to track the precision and accuracy of the laboratory's analyses, and to identify areas that require or could benefit from improvement.

16.1.3 The following types of QC analyses are performed on a scheduled basis at the laboratory:

16.1.3.1 Re-analysis of client samples by the same analyst (self-check analysis) or by a different analyst (cross-check analysis).

16.1.3.2 Repeated analyses on calibration standards of known asbestos concentration.

16.1.3.3 NIST proficiency testing.

16.1.3.4 Inter-laboratory analyses.

16.1.4 Records are kept of all QA documentation.

16.1.5 All QC analyses must be performed in real-time.

16.2 Calibration Standards

16.2.1 Visual estimates of asbestos concentrations are calibrated with the use of the calibration standards.

16.2.2 The calibration standards are a set of permanently mounted slides of known asbestos concentrations. They should cover a wide range of asbestos concentrations.

16.2.3 Reference materials used to prepare calibration standards are NIST SRM's 1866b and 1867a, Controlled PE Reference Materials, and samples from past NIST proficiency testing rounds.

16.2.3.1 Controlled PE Reference Materials at concentrations of 0.2% and 1.0% LA in soils are required to delineate between the bin assignments; however, those concentrations, as well as concentrations of 0.5% and 2.0%, are useful for the calibration of visual area estimates for low end samples.

16.2.3.2 "Working standard" refers to any calibration standard that was prepared internally at the laboratory. Laboratories are encouraged to prepare these standards over a range of asbestos concentrations. These slides should not just be prepared of Libby Amphibole but for other asbestos types as well.

16.3 Use of Calibration Standards for Precision and Accuracy Testing

16.3.1 The best way to track analyst precision and accuracy is by the analysis of standards of known asbestos concentration.

16.3.1.1 All analysts need to analyze calibration standards on a regular basis.

16.3.1.2 Regular analysis of the calibration standards provides a routine check of analyst precision and accuracy.

16.3.1.3 Calibration standards are read at a frequency on one per 100 client samples.

16.3.2 Vary the calibration standards read each month so that analysts are constantly presented with standards of different asbestos concentrations, various asbestos types, and various matrix material types.

16.3.3 The analysts must be blind to the known values of the calibration standards.

16.3.4 The Laboratory Manager, QA/QC Coordinator, or designate other than the analyst performing the test, will review the results for acceptability.

16.3.5 After completion of analyses of calibration standards, analysts are advised of the reference values of the standards so they can see how they performed and calibrate their readings on client samples accordingly. For example, the reported value of blind calibration standards below 1% should fall in the correct concentration bin.

16.3.6 Repeated analysis of the calibration standards provides a benchmark upon which analysts may base their visual estimations of percentage levels of asbestos in client samples. Use of control charts for concentrations 1% or greater is recommended.

16.3.7 Corrective action(s) must be taken immediately if calibration standards do not meet acceptance criteria. Examples of corrective actions that may be taken are re-analysis of calibration standards, re-preparation of calibration standards, and analyst re-training.

16.3.8 Analyses of the calibration standards are not reported as part of an EDD or data package. Rather, laboratories are responsible for maintaining an internal system for tracking analyses of these calibration standards.

16.4 Self-Check and Cross-Check QC Analyses (Duplicates and Replicates)

16.4.1 For each set of samples, 10% of the samples must be re-analyzed within the laboratory.

16.4.2 A QC analysis (self-check or cross-check) can be performed on any sample.

16.4.2.1 QC analyses need to be performed on samples over the entire range of asbestos concentrations that are encountered in site samples.

16.4.2.2 Any sample that is considered especially unusual or difficult should be re-analyzed for QC purposes.

16.4.3 The frequency of self-check QC analyses on client samples will be 1 per 50 samples analyzed (2%). Self-check analyses should be performed as a remount of the sample (see Section 13.3 for slide preparation procedures).

16.4.4 The frequency of cross-check QC analyses on client samples will be 8 per 100 samples analyzed (8%). Cross-check analyses should be done on the five original slide preparations.

16.4.4.1 All analysts performing QC analyses must be experienced with PLM analysis of soil samples from the Libby Asbestos Superfund Site and the specific requirements of this SOP.

16.4.4.2 If there is only one primary analyst at the laboratory performing PLM analysis on these samples, the laboratory must send all cross-check QC samples to another Libby laboratory with the proper experience and qualifications.

16.4.5 The self-check and cross-check analysis is acceptable if results are within a bin

category (i.e., ± 1 bin) for reported concentrations below 1% LA. For all asbestos types greater than 1%, it is recommended that precision is tracked using control charting or a similar tool.

16.4.6 Corrective action(s) must be taken immediately if re-analyses do not meet acceptance criteria. Examples of corrective actions that may be taken are re-analysis and/or re-preparation and re-analysis of original and duplicate or replicate samples, analyst re-training, and notification to EPA, or their designate.

16.4.7 When performing a QC analysis, it is necessary to mark LDS or LDC in the "QA Type" section of the bench sheet.

16.5 Inter-Laboratory Analyses

16.5.1 The laboratory is involved in an ongoing sample exchange program with other PLM laboratories that analyze soil samples from the Libby Asbestos Superfund Site. The purpose of this program is to help detect and minimize laboratory biases and characterize precision across laboratories performing PLM-VE testing.

16.5.2 The frequency of the inter-laboratory sample exchange ranges from 1 in 100 samples analyzed exchanged amongst laboratories on a quarterly basis. However, higher frequencies of inter-laboratory sample analysis are required when a laboratory is new to the program, when systematic errors or biases are observed, or when a new version of the SOP is distributed. Whether or not the frequency to be performed is the minimum or higher is determined by EPA or their designate.

16.5.3 Results of the inter-laboratory analyses are reviewed by EPA, or their designate.

16.5.4 The inter-laboratory analysis is acceptable if results are within a bin category (i.e., ± 1 bin) for reported concentrations below 1% LA.

16.5.5 Corrective action(s) must be taken immediately if analyses do not meet acceptance criteria. The specific course of action based on these results will be determined by EPA, or their designate. Common actions include re-analysis and/or re-preparation and re-analysis of original and duplicate or replicate samples, collaboration between and amongst laboratories performing the test to root out biases, and analyst re-training.

17.0 REFERENCES

17.1 Bandli, B.R. et al. (2003). Optical, compositional, morphological, and X-ray data on eleven Eleven particles of amphibole from Libby, Montana, U.S.A. Canadian Mineralogist, 41, 1241-1253.

18.2 Bloss, F.D. (1999). Becke Line Chart. Optical Crystallography. Mineralogical Society of America, Monograph Series Publication No. 5.

18.3 Cornelis Klein and Cornelius S. Hurlbut, Jr. (1985). Manual of Mineralogy. John Wiley & Sons.

18.4 Deer, W.A., Howie, R.A., and Zussman, J. (1997). Rock Forming Minerals Volume 2B: Double Chain Silicates. 2nd Edition. The Geological Society, London.

18.5 EPA Method 600/R-93/116: Method for the Determination of Asbestos in Bulk Building Materials. (July 1993).

18.6 Federal Register, 40 CFR Part 763, Volume 52, No 210, "Asbestos Containing Materials in Schools; Final Rule and Notice."

18.7 McCrone Research Institute (1987). Dispersion Staining Color Chart.

18.8 Meeker, G.P., Bern, A.M., Brownfield, I.K., Lowers, H.A., Sutley, S.J., Hoefen, T.M., and Vance, J.S. (2003). The Composition and Morphology of Amphiboles from the Rainy Creek Complex Near Libby Montana. American Mineralogist, 88, 1955-1969.

18.9 NIOSH Method 9002: Asbestos (bulk) by PLM. Manual of Analytical Methods, 4th Edition. (August 1994).

18.10 NIST Handbook 150, 2006 Edition, "NVLAP Procedures and General Requirements."

18.11 State of California Air Resources Board Method 435: Determination of Asbestos Content of Serpentine Aggregate. (June 1991).

18.12 Su, Shu-Chun (2004). Area Percentage Charts With Matrix and Area Percentage Charts With No Matrix.

18.13 Su, Shu-Chun (1996). Rapidly and Accurately Determining Refractive Indices of Asbestos Fibers by Using Dispersion Staining Method.

18.14 Terry and Chilingar (1955). Comparison Chart for Visual Percentage Estimation.

18.15 William D. Nesse (1991). Introduction to Optical Mineral Mineralogy. New York: Oxford University Press.

18.16 Wylie, A.G. and Verkouteren, J.R. (2000). Amphibole asbestos from Libby, Montana: Aspects of nomenclature. American Mineralogist, 85, 1540-1542.

19.0 LIST OF ATTACHMENTS

19.1 Attachment 1: PLM-VE Data Recording Sheet

19.2 Attachment 2: RI Liquid Calibration Conversion Tables

19.3 Attachment 3: Data Package Checklist from PLM Data Sheet and EDD

19.4 Attachment 4: Optical Properties of Fibrous Amphiboles

19.5 Attachment 5: PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Morphologies

19.6 Attachment 6: SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

19.7 Attachment 7: Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Controlled PE Reference Materials

19.8 Attachment 8: Flow Diagram for Determining LA Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

ATTACHMENT 1

PLM-VE Data Recording Sheet

PLM VISUAL ESTIMATION DATA RECORDING SHEET

Laboratory Name ________________________________ Date Received ____________

Job Number ________________________________ SOP Name/Revision ____________________

Note: Data Recording Sheet is formatted to print on 11x17 paper.

EPA Index ID	Index Suffix Char.	Index Suffix No.	QA Type (NOT QA, LDS, LDC)	Lab Sample ID	Date Analyzed	Analyst Name	Stereomicroscopy Examination					Libby Amphibole		Other Amphibole			Chrysotile		Deviation?	Comments (list below)	OPTICAL PROPERTIES FOR LA (see key for appropriate data inputs)							
								Est. % LA		Est. % OA and C																		
							Sample Appearance	Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	Qual (ND, Tr, <)	Mass Fract (%)	Qual (ND, <)	Area Fract (%)	OA Type (AMOS, ANTH, CROC, UNK)	Qual (ND, <)	Area Fract (%)			Morph.	Fiber Color	Sign Elong. (+/-)	Pleoch. (Y/N)	Angle Extinct.	Ref. Index α	Ref. Index γ	Biref.

Comments (Use back if needed)

ATTACHMENT 2

RI Liquid Calibration Conversion Tables

Prepared by Dr. Shu-Chun Su, Hercules, Inc.

See attached Excel spreadsheet entitled

"Create_RI_Liquid_Calibration_Conversion_Tables.xls"

ATTACHMENT 3

Data Package Checklist

From PLM (VE and PC) Data Sheet and EDD.xls

STANDARD LABORATORY DATA PACKAGE CHECKLIST
Analytical Test Report
Bulk Asbestos Analysis by Polarized Light Microscopy (PLM)

Prepared For:

City/State: _____

Laboratory Name: _____

City/State: _____

Laboratory Job No.: _____

Method Utilized (SOP and Rev. No.): SRC-LIBBY-03/Revision 2

Circle One:

Visual Estimation Point Counting Approach

Report Reviewed by: _____

STANDARD LABORATORY DATA PACKAGE CHECKLIST

	Instructions: For PLM analytical results raw data packages, complete and sign the following checklist. Attach supporting documentation as outlined below. Organize the supporting documentation in the order listed below. Paginate the completed raw data package.	Laboratory Verification (Initials and Date)	Validator Verification (Initials and Date)
1	**Number of samples received:**_____ *An SDG is defined as no more than 200 samples.* **Additional Supporting Documentation:** Attach COC forms having footer R (report).		
2	**Date of sample receipt and condition of samples:**_____ *For Condition of samples enter "OK" or "See SDG Case Narrative".*		
3	**SDG Case Narrative:** **Additional Supporting Documentation:** Attach SDG Narrative and any modification forms.		
4	**Check for contamination (daily):** Wipe microscope slides with lens paper before using. *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
5	**Verification of the refractive indices of the refractive index liquids once per month:** **Additional Supporting Documentation:** Provide information indicating a monthly record of checking each of the four liquids including liquid name, lot number and analyst initials. (See table - Results of RI Liquids Calibration)		
6	**Verification of microscope adjustments prior to each SDG:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
7	**Reference material - Visual Estimation Approach:** *Laboratory Verification initial and date signifies that this has been performed for the samples in this SDG.*		
	Reference material - Point Counting Approach: **Additional Supporting Documentation:** Provide calibration curve documentation, printed from the EDD spreadsheet.		
8	**VE and/or PC hard copy data forms (as presented in the EDD spreadsheet):** **Additional Supporting Documentation:** Copies of the Hard Copy Data Forms for all investigative samples and laboratory duplicates will be provided from systems that are entered electronically.		
9	**Bench sheets for data results:** **Additional Supporting Documentation:** Provide copies of the hand written or LIMS system generated raw data sheets for sample results.		

COCs

SDG NARRATIVE

Instructions: The following information should be included in all narratives. Please see the attached narrative template.

1 List the method or methods used.
2 For any modifications, reference the modification number and attach a copy of the signed document to the raw data
3 If sample condition is not "OK", explain why and any implications to the data.

SDG NARRATIVE EXAMPLE

SDG Narrative - PLM Analysis by SRC-LIBBY-03 Revision 2

Laboratory Job Number:_____

The samples were received in sealed coolers [or other container]. [Any special notations about the samples as received goes here such as damaged in shipping, missing sample, etc.] The sample set was assigned a laboratory job number, each sample was assigned a unique, sequential laboratory ID number, and the job was entered into the Laboratory Information System. The laboratory ID numbers, shipping information and signatures were recorded on the CDM Chain of Custody and the login information was summarized on the laboratory Chain of Custody.

Samples were analyzed in accord with SRC-LIBBY-03 Rev. 2 [with modifications described on Laboratory Modification document(s): LB-_____ (see attached)].

SAMPLE RESULTS

See Attached Sample Results

Instructions: These sample result forms are from the current version of the PLM (VE & PC) Data Sheet and EDD.xls file. They are labeled in this file as the VE or PC hard copy data form.

BENCH SHEETS

Instructions: Please provide handwritten or LIMS system generated raw data sheets for sample results.

REFRACTIVE INDEX LIQUIDS

Instructions: Please see and follow attached table from Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs. (Suggested Format for Recording Results of RI Liquids Calibration using Cargille Glass Standard and Dispersion Staining Method - Version: February 1996)

The following components are included in the table:

1 Date
2 Nominal or Labeled n_D 25 degree Celsius
3 Cargille Glass
3a Nominal or Labeled R.I.
3b Lot No.
4 Central Stop DS Observation
4a Predominant DS Color
4b Corresponding $alpha_0$
5 Liquid or Room Temperature (degree Celsius)
6 Actual or Calibrated n_D 25 degree Celsius
7 Difference between Calibrated n_D 25 degree Celsius and Labeled n_D 25 degree Celsius
8 Accept or Reject
9 Analyst

RESULTS OF RI LIQUIDS CALIBRATION

Date[1]	RI Liquid[2]	Cargille Glass		CSDS Color of Glass4		Liquid or Room Temperature (°C)[7]	Calibrated RI of Liquid $(n_D^{25°C})_{cdb}$[8]	Absolute Difference Between Calibrated and labeled RI $(n_D^{25°C})_{cdb} - (n_D^{25°C})_{lbl}$[9]	Accept or Reject[10]	Initials of Analyst[11]
	Labeled RI $(nD25°C)_{lbl}$	Labeled RI[3]	Lot No.[4]	Predominant CSDS Color[5]	Corresponding λ_0 (nm)[6]					
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	
									A R	

1. Date: 2. The $n_D^{25°C}$ on the label of RI liquid bottle or $(n_D^{25°C})_{lbl}$; 3. The RI value on the label of Cargille calibrated glass vial; 4. The Lot No. on the label of Cargille calibrated glass vial; 5. The predominant central stop dispersion color displayed by glass fragments (do not be confused by the false CSDS color due to edge effect (see p.3). 6. The matching wavelength, λ_0, corresponding to the CSDS color in Column 5; 7. The temperature of the RI liquid or the room if the liquid's temperature can be considered to be in equilibrium with the room atmosphere; 8. The reading based on the values in Columns 6 and 7 from the conversion table for the liquid-glass combination. This value is the actual or calibrated RI of the liquid at 589 nm and 25 °C or $(n_D^{25°C})_{cdb}$; 9. Column 8 minus Column 2; 10. If the *absolute* value of Column 9 is less or equal to 0.004, circle A for *acceptable*. Otherwise, circle R for *rejected*. 11. Analyst's initials.

$(nD25°C)_{lbl} = n_D^t + (25 - t)\, dn/dt$ (The temperature correction is built in the conversion tables.)

Version: December 1998 (Shu-Chun Su, Technical Expert for NVLAP Asbestos Programs)

ATTACHMENT 4

Optical Properties of Fibrous Amphiboles

OPTICAL PROPERTIES OF FIBROUS AMPHIBOLES ASSOCIATED WITH LIBBY AMPHIBOLE[A]

Libby Amphibole asbestos (LA) is a term used to categorize a group of minerals generally described as sodic tremolite. The solid solution series of sodic tremolite is comprised of a group of minerals, such as tremolite, actinolite, winchite, richterite, magnesio-riebeckite, and magnesio-arfvedsonite. The optical properties for each individual mineral are provided below. As seen, there is a great deal of overlap in optical properties among the minerals that make up LA. As such, discreet mineral identification is not required under this SOP. Rather, if the sample exhibits the optical properties of a mineral listed below, the specific optical properties (refractive indices, birefringence, extinction angle, and elongation sign) shall be noted on the analytical data sheet and electronic file, and the mineral identified as LA.

Mineral	Morphology and Color	Refractive Indices		Birefringence	Extinction	Elongation Sign
		α	γ			
Tremolite[7]	Straight to curved fibers and bundles. Colorless to pale green.	1.600-1.628 1.604-1.612 1.599-1.612 1.6063	1.625-1.655 1.627-1.635 1.625-1.637 1.6343	0.017-0.028	Oblique (up to 21 °);	+ (length slow)
Actinolite[7]		1.600-1.628 1.612-1.668 1.613-1.628 1.6126	1.625-1.655 1.635-1.688 1.638-1.655 1.6393	0.017-0.028		+ (length slow)
Winchite	Straight to curved fibers or bundles. Colorless to pale blue Pleochroism weak to moderate: X-colorless, Y=light blue-violet, Z=light blue[3]	**1.618-1.626**[1] **1.618-1.621**[2] 1.629[3] 1.636[4]	**1.634-1.642**[1] **1.634-1.637**[2] 1.650[3] 1.658[4]	**0.008-0.019**[1] **0.016**[2] 0.021[3] 0.022[4]	Oblique, **22°**[1] **15.8°**[2] Oblique, 7-29°[8]	+ (length slow)
Richterite	Straight to curved fibers or bundles. Colorless, pale yellow, brown, pale to dark green, or violet[8] Pleochroism weak to strong in pale yellow, orange, and red[5]	**1.622-1.623**[1] 1.605-1.624[5] 1.615[6]	**1.638-1.639**[1] 1.627-1.641[5] 1.636[6]	**0.012-0.017**[1] 0.017-0.022[5]	Oblique, **21-22°**[1] Oblique, 5-45°[8]	+ (length slow)
Magnesio-riebeckite	Prismatic to fibrous aggregates. Blue, grey-blue, pale blue to yellow. Can be pleochroic.[8]	1.650-1.673[8]	1.662-1.676[8]	Up to 0.015[8]	Oblique, 8-40°[8]	- (length fast)[8]
Magnesio-arfvedsonite	Prismatic to fibrous aggregates. Yellowish green, brownish green, or grey-blue. Can be pleochroic.[8]	1.623-1.660[8]	1.635-1.680[8]	0.012-0.026[8]	Oblique, 18-45°[8]	- (length fast)[8]

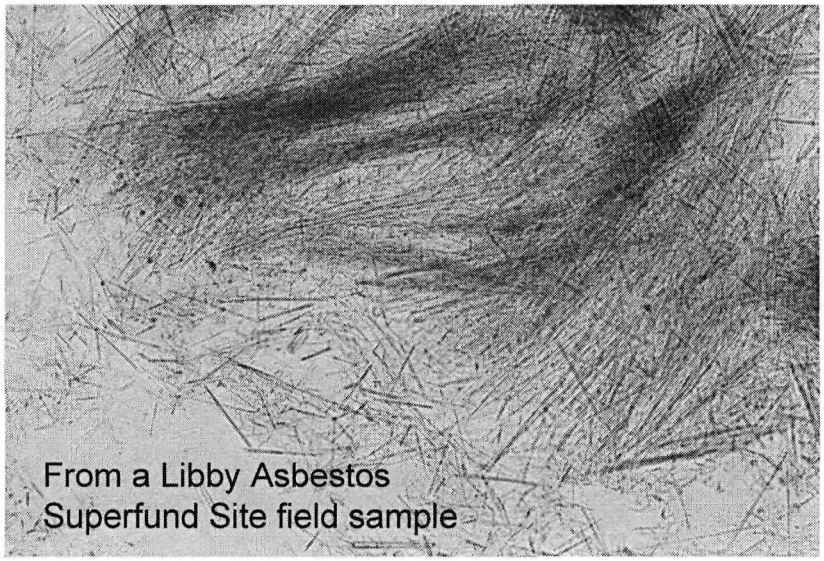
A. This table is adapted for use in the SOP from: Su, Shu-Chun, 2005. White paper: *Tables to Facilitate the Determination of Refractive Indices of Winchite and Richterite, (Libby, Montana) by Dispersion Staining*, **August 8, 2005** Data on this table were compiled from data of amphiboles from Libby, Montana and other localities. The data in **bold** are samples from Libby, Montana. The data of tremolite/actinolite are adapted from Table 2-2 of EPA/600/R-93/116.

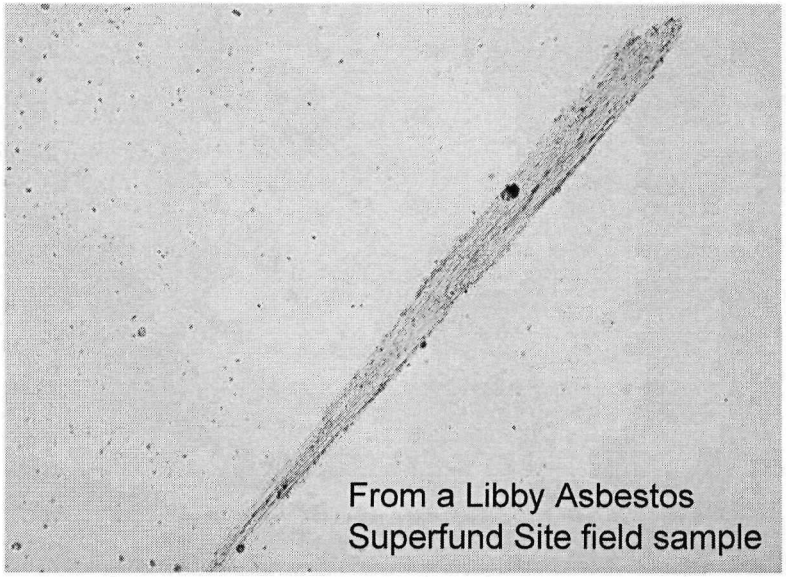
1. Bandli, B.R. et al. (2003) *Optical, compositional, morphological, and X-ray data on eleven particles of amphibole from Libby, Montana, U.S.A.* Canadian Mineralogist, 41, 1241-1253.
2. Wylie, A.G. and Verkouteren, J.R. (2000) *Amphibole asbestos from Libby, Montana: Aspects of nomenclature.* American Mineralogist, 85, 1540-1542.
3. www.minsocam.oeg/msa/Handbook/Winchite.PDF.
4. www.mindat.org/min-4296.html.
5. www.minsocam.oeg/msa/Handbook/Richterite.PDF.
6. www.webmineral.com/data/Richterite.shtml.
7. Adapted from: USEPA 1993. *Method for the Determination of Asbestos in Bulk Building Materials*. July 1993. (NTIS / PB93-218576).
8. W. A. Deer, R. A. Howie, and J. Zussman (1997). *Rock Forming Minerals Volume 2B: Double Chain Silicates, 2nd Edition.* The Geological Society, London. Optical properties for magnesio-riebeckite and magnesio-arfvedsonite inserted by Douglas Kent at ESAT Region 8, October 2008.

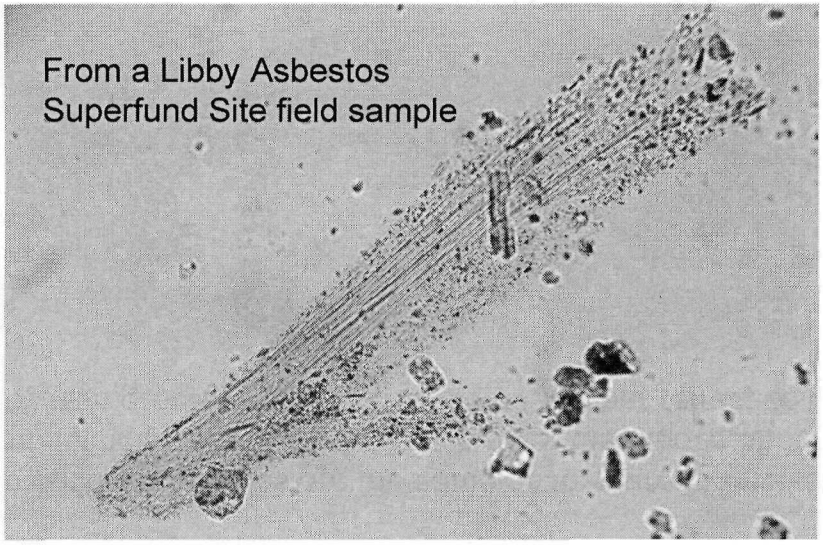
ATTACHMENT 5

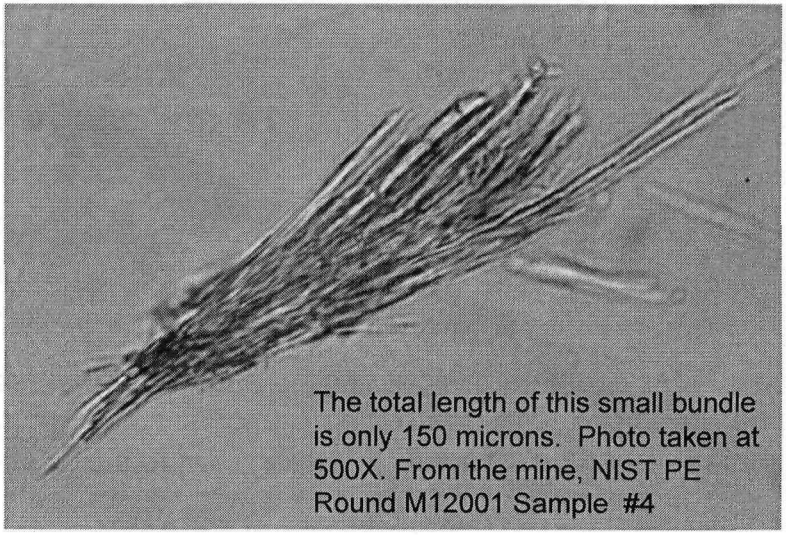
PLM Photomicrographs Demonstrating a Wide Range of Libby Amphibole Morphologies

PLM Photomicrographs of Typical Libby Amphibole Morphology



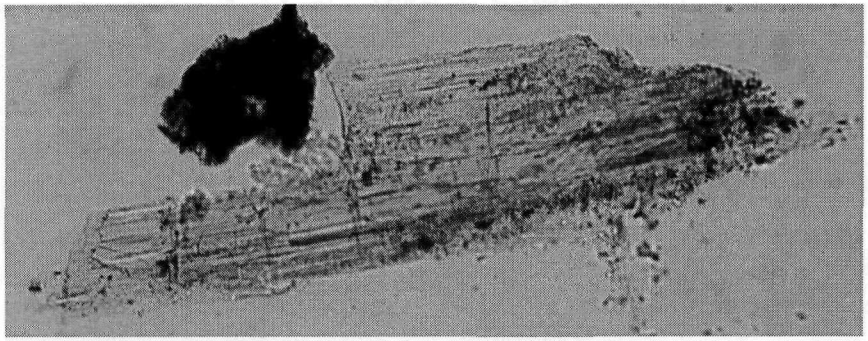




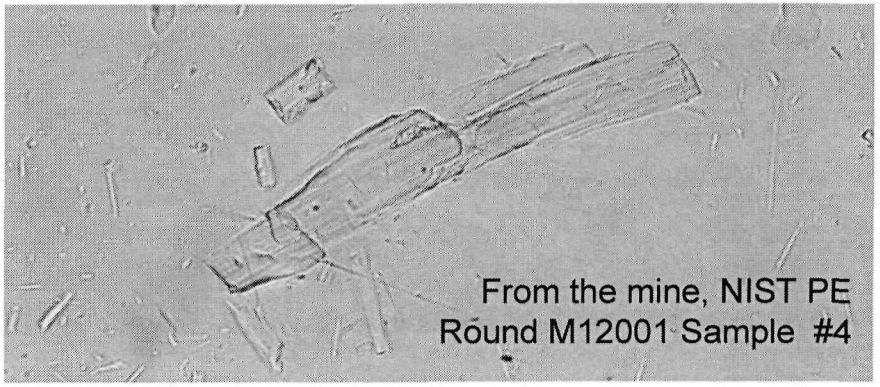


Prismatic Libby Amphibole

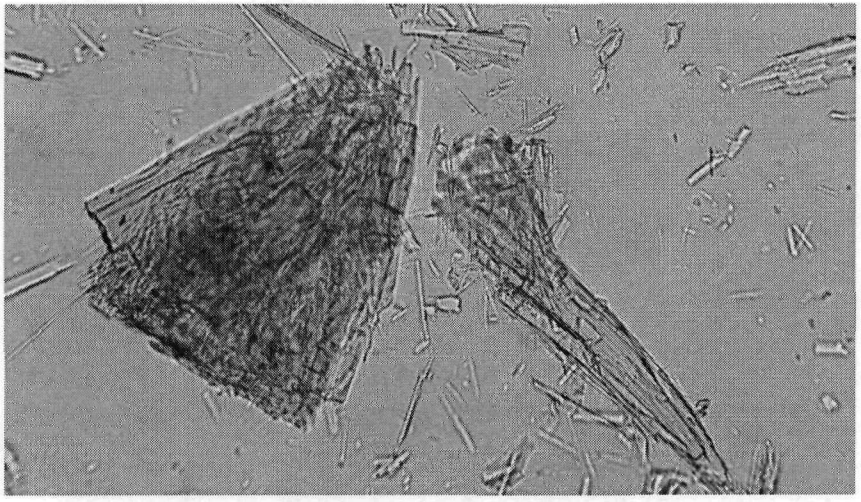
The optical properties are the same as they are for more fibrous forms of LA. Colors of winhcite, richterite, tremolite, and actinolite are generally much paler than those of hornblende, which is usually dark green to dark blue-green to brownish green. Hornblende also has higher refractive indices (in the range of 1.65 to 1.68) than Libby Amphibole.



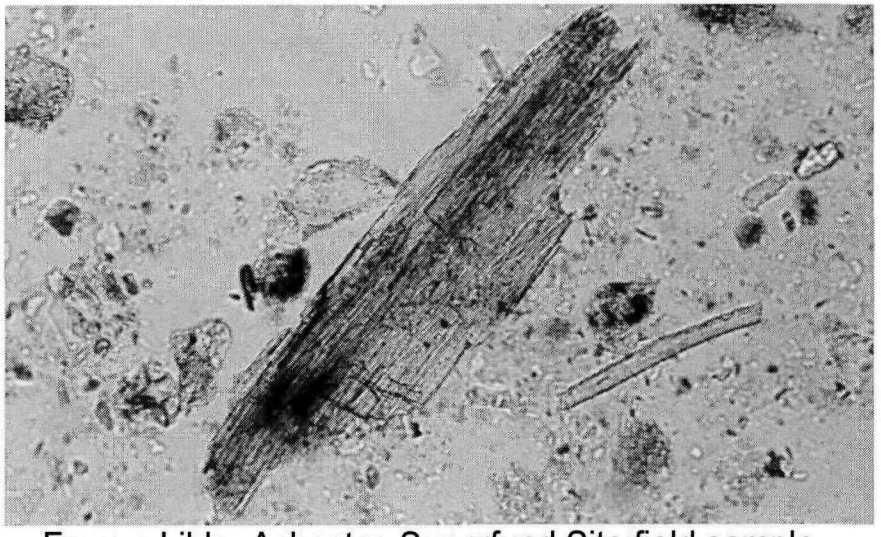
From a Libby Asbestos Superfund Site field sample



From the mine, NIST PE Round M12001 Sample #4

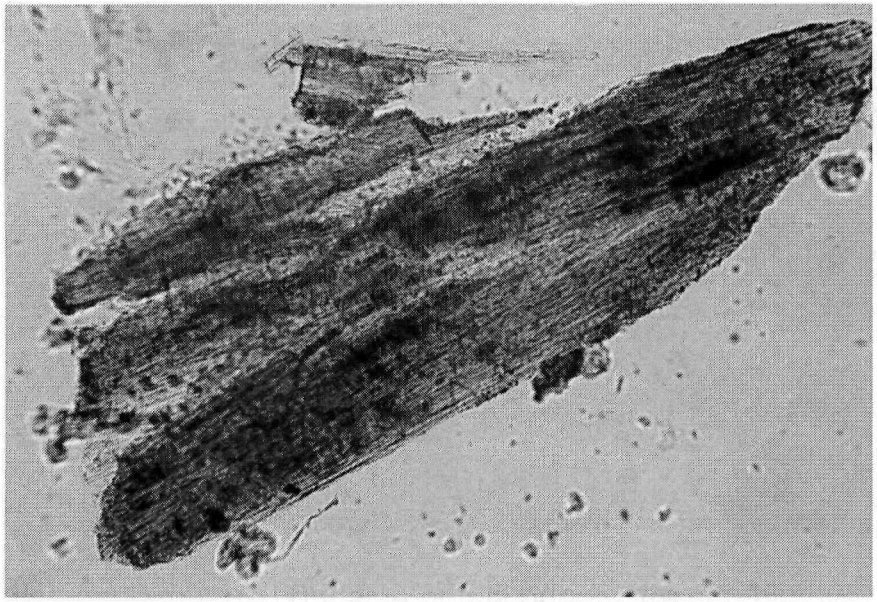


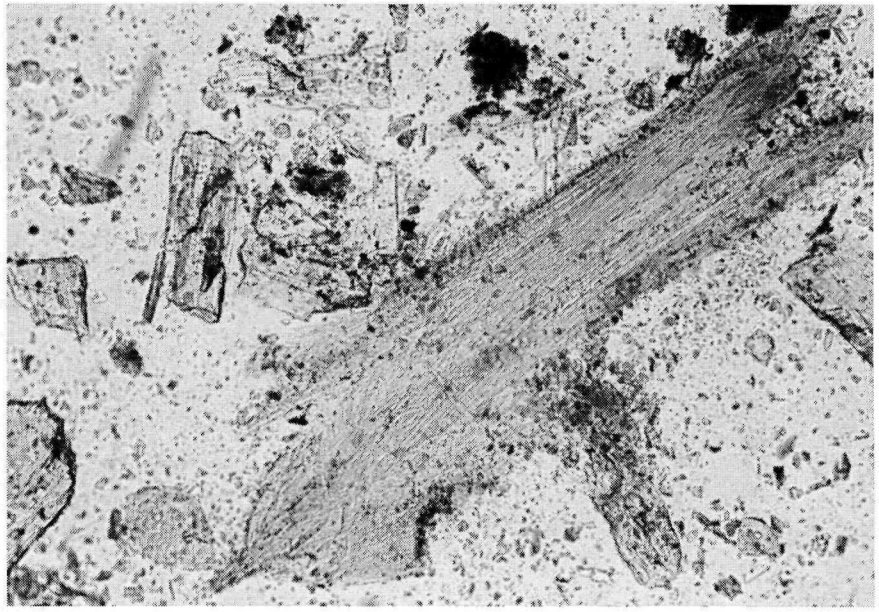
From the mine, NIST PE Round M12001 Sample #4

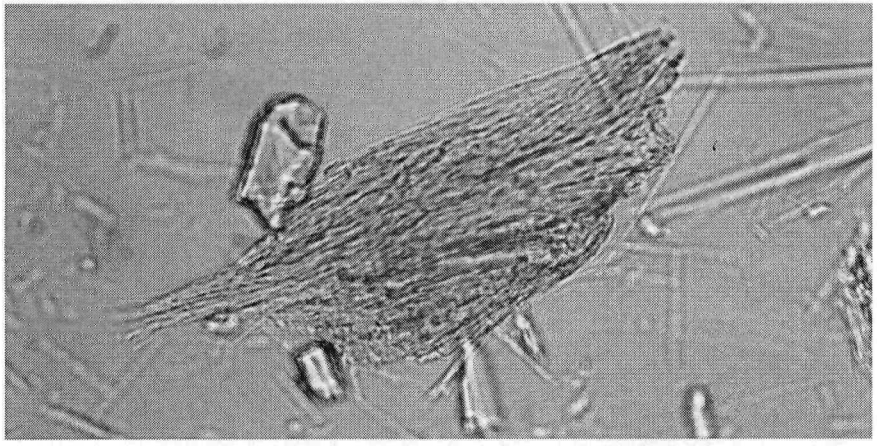


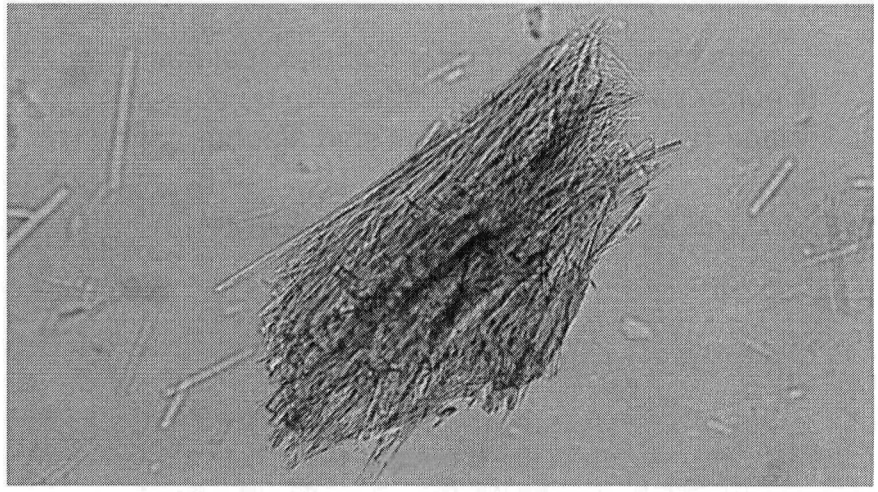
From a Libby Asbestos Superfund Site field sample

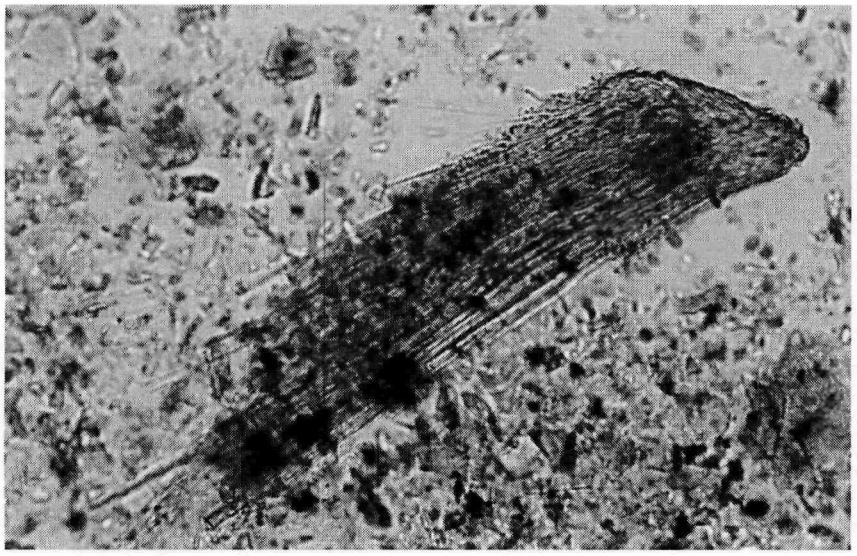
Some Libby Amphibole shows a "matted" or "felted" morphology. The internal structure of these bundles is still fibrous. The green high-relief prismatic crystals in the top right photo are hornblende. The bundles in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles in the lower two photos are from the NIST PE Round M12001 Sample #4, from the mine.



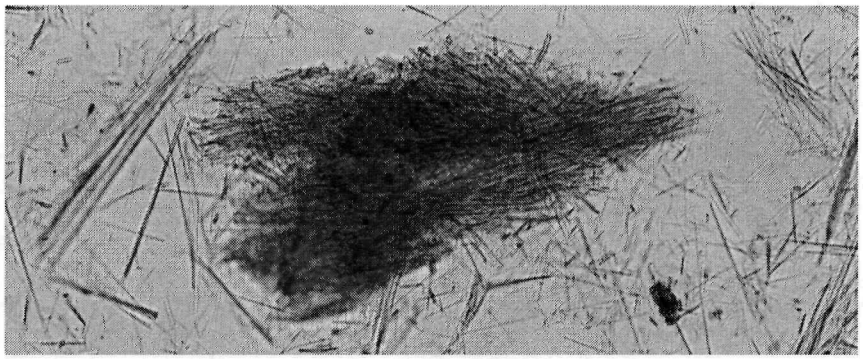




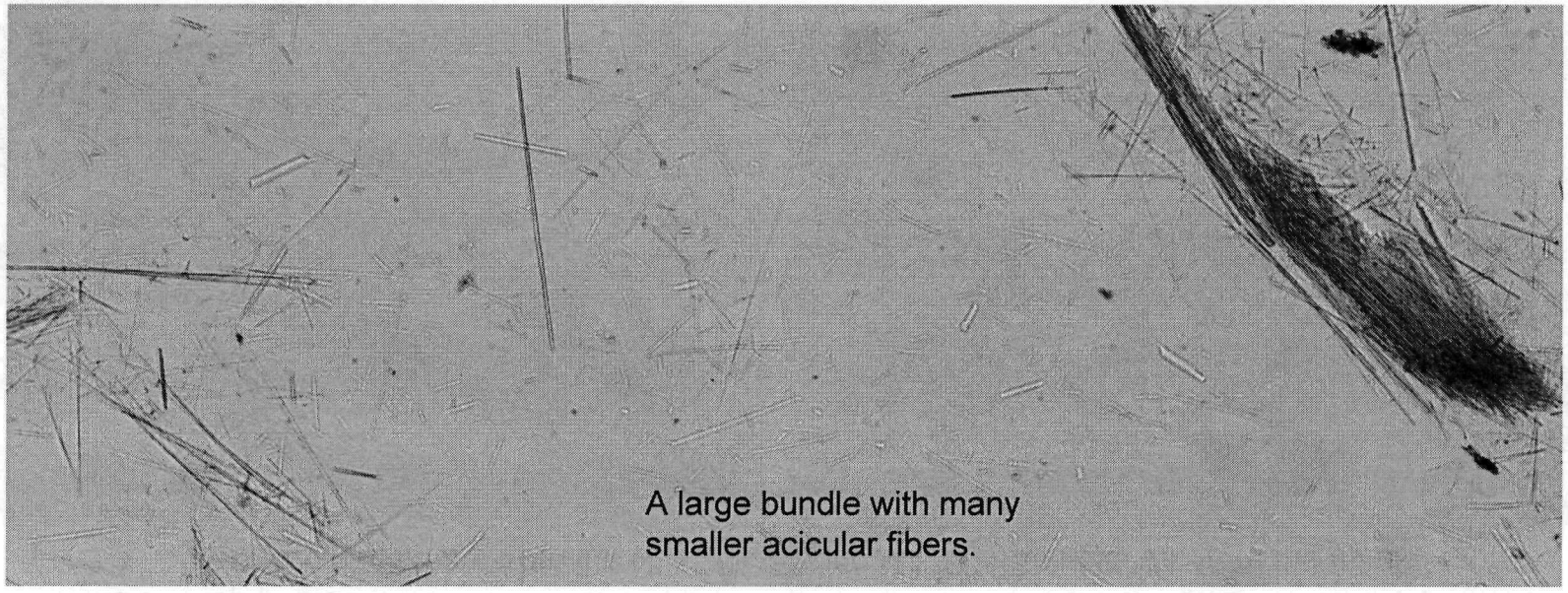




The fibers on the right side of this bundle are completely matted.

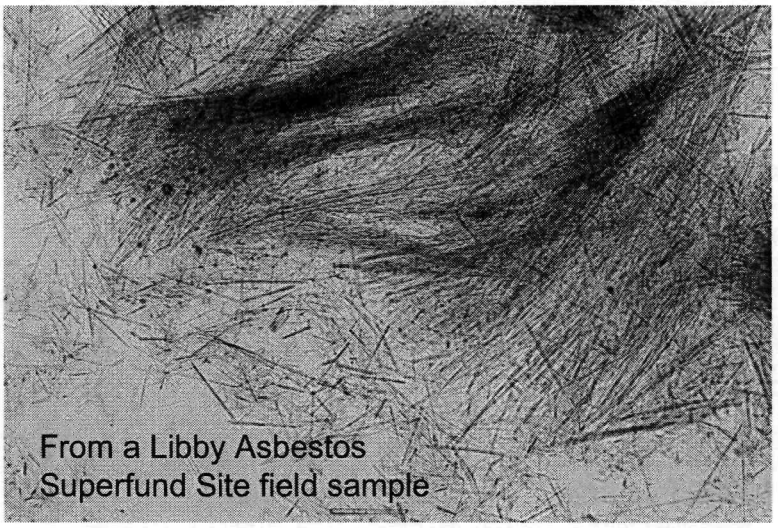


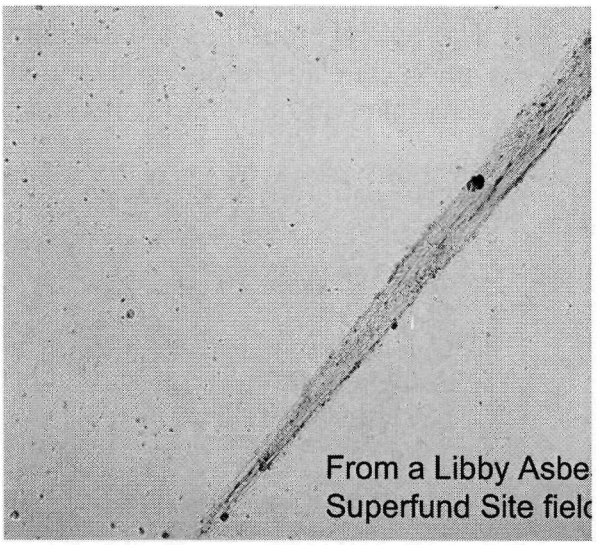
A "felted" bundle plus some smaller acicular fibers. The photos on this page are all of bundles found in field samples collected from the Libby Asbestos Superfund Site.

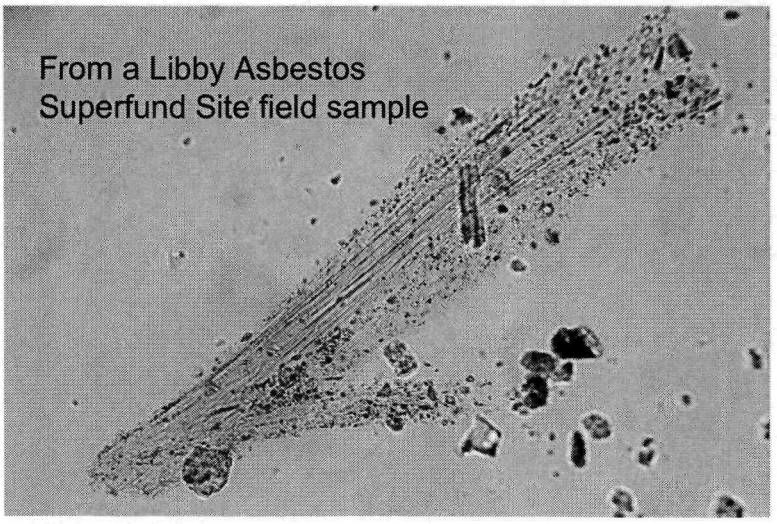


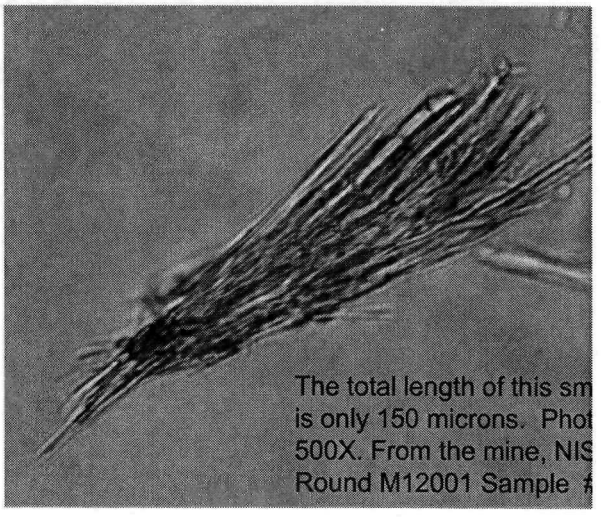
A large bundle with many smaller acicular fibers.

PLM Photomicrographs of Typical Libby Amphibole Morpholog



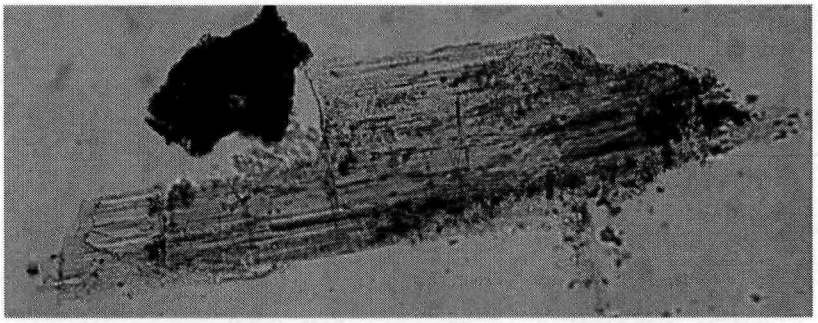




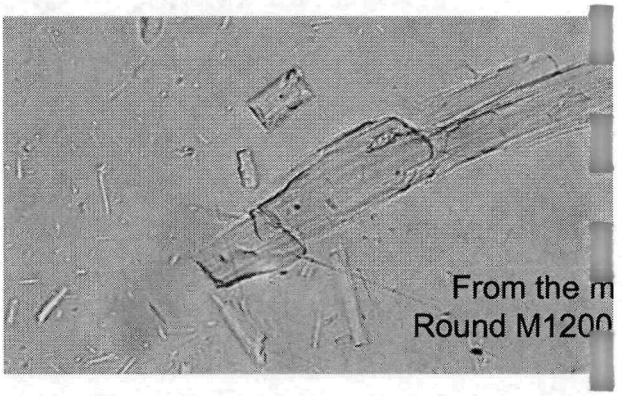


Prismatic Libby Amphibole

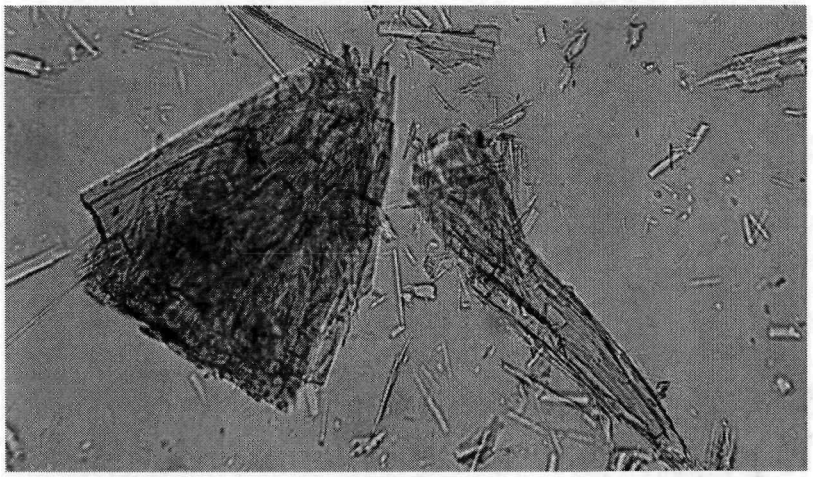
The optical properties are the same as they are for more fibrous forms of LA. Colors of winhcite, richteri and actinolite are generally much paler than those of hornblende, which is usually dark green to dark brownish green. Hornblende also has higher refractive indices (in the range of 1.65 to 1.68) than Libby



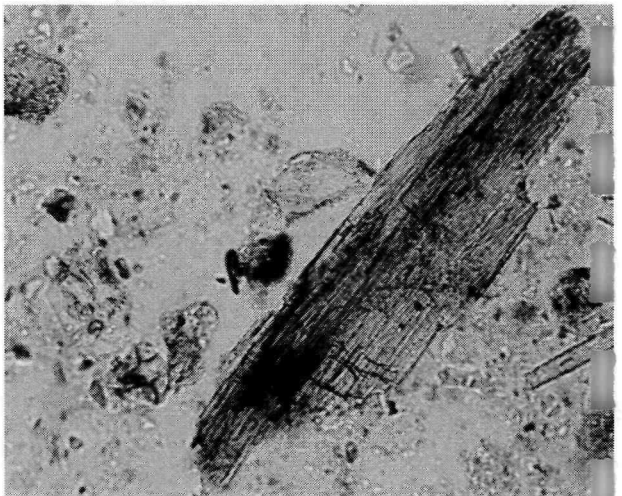
From a Libby Asbestos Superfund Site field sample



From the m
Round M120

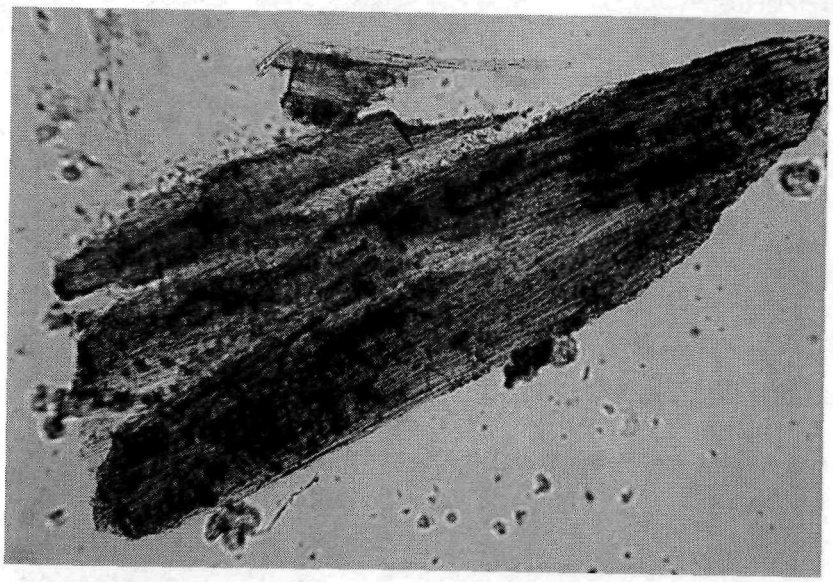


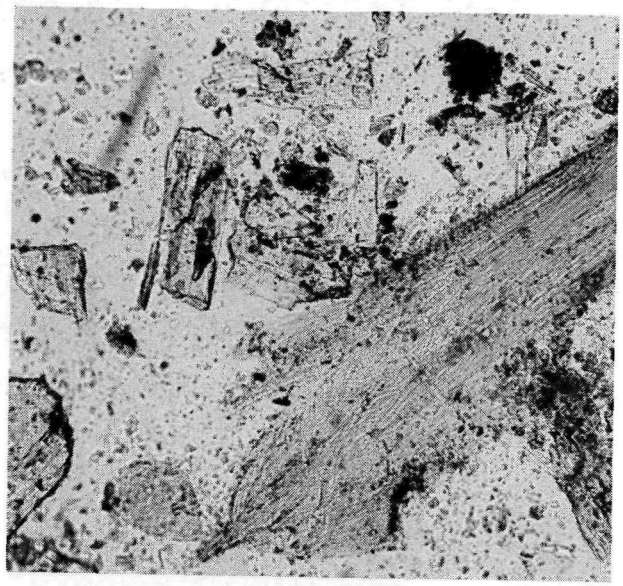
From the mine, NIST PE Round M12001 Sample #4

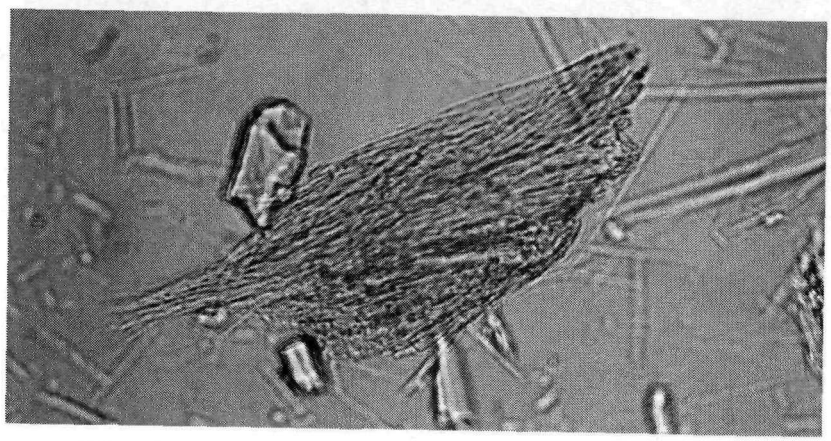


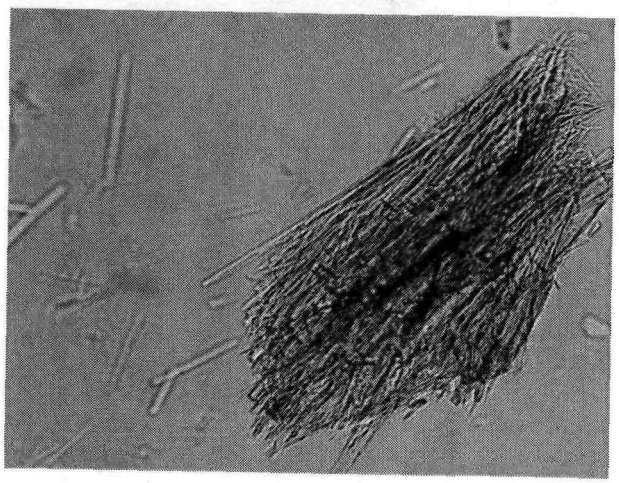
From a Libby Asbestos Superfund Site

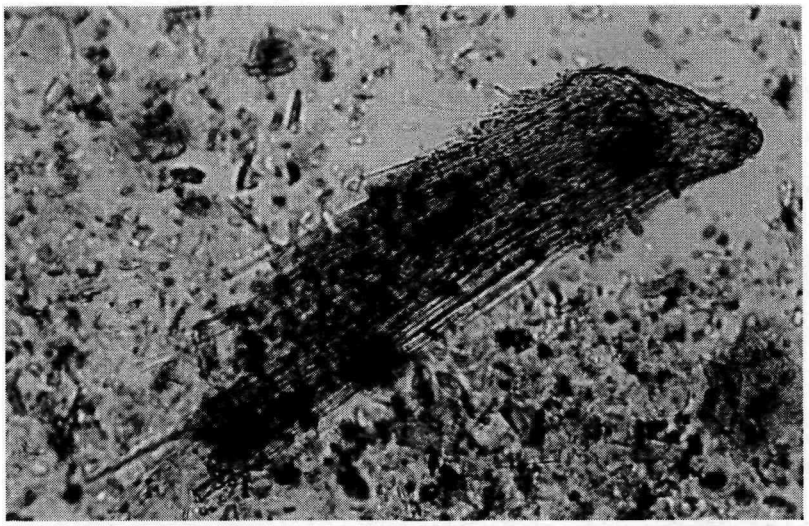
Some Libby Amphibole shows a "matted" or "felted" morphology. The internal structure of these is still fibrous. The green high-relief prismatic crystals in the top right photo are hornblende. The in the two top photos were found in Libby Asbestos Superfund Site field samples. The bundles lower two photos are from the NIST PE Round M12001 Sample #4, from the mine.



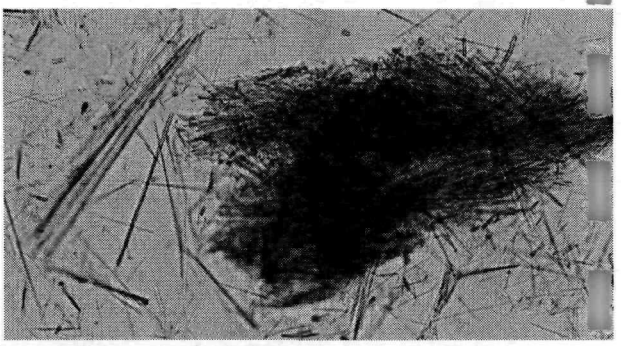




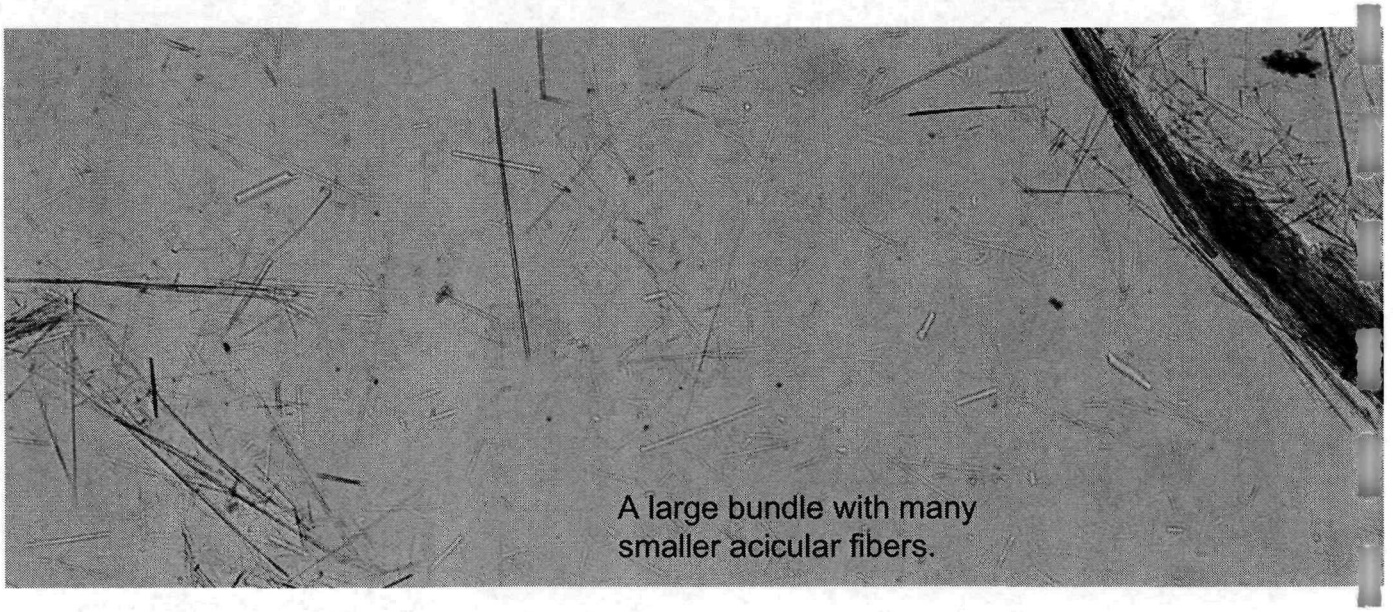




The fibers on the right side of this bundle are completely matted.



A "felted" bundle plus some smaller aci
The photos on this page are all of bun
field samples collected from the Libby A
Superfund Site.



A large bundle with many
smaller acicular fibers.

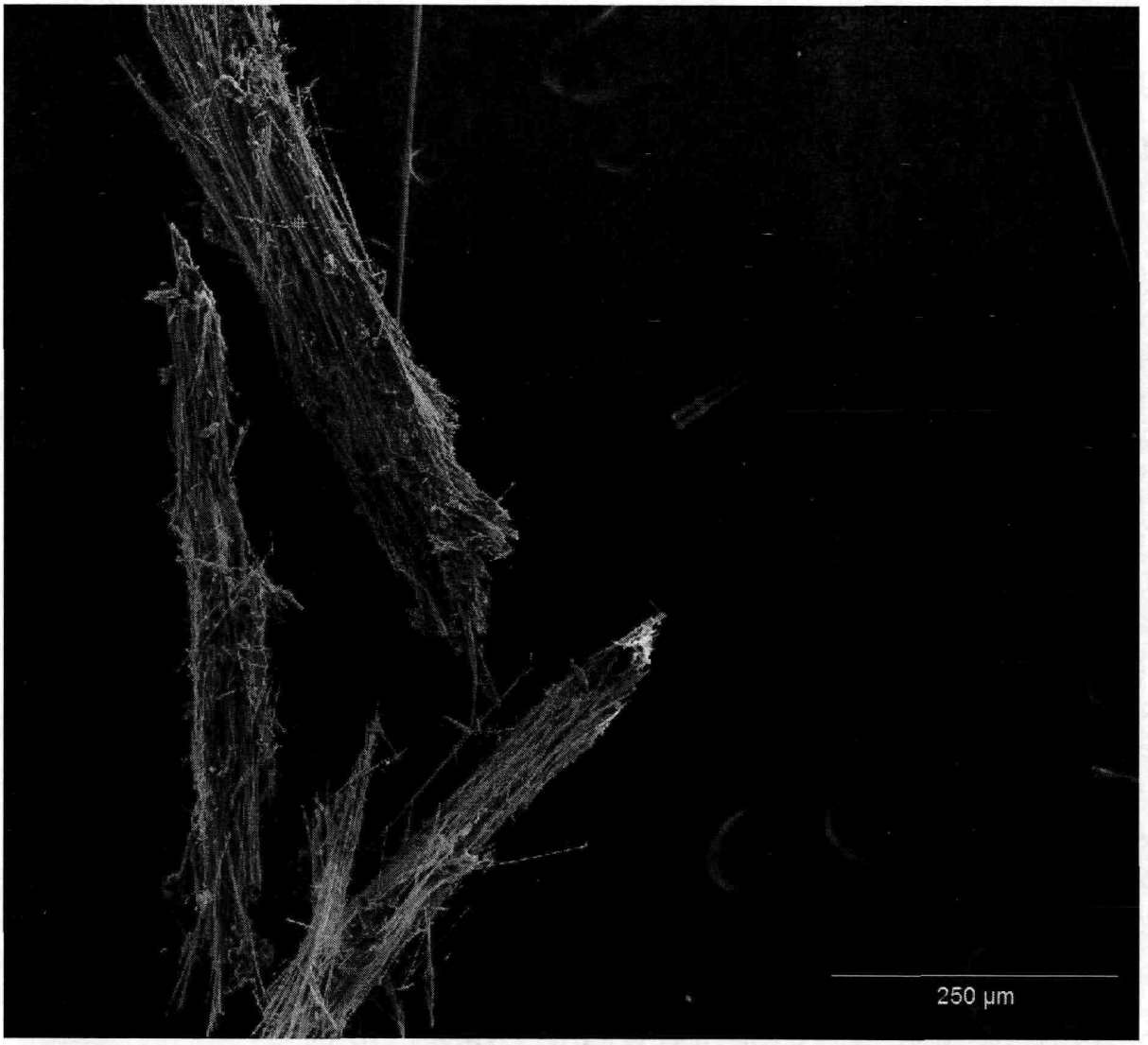
ATTACHMENT 6

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphologies

SEM Photomicrographs of Representative Examples of Libby Amphibole Morphology

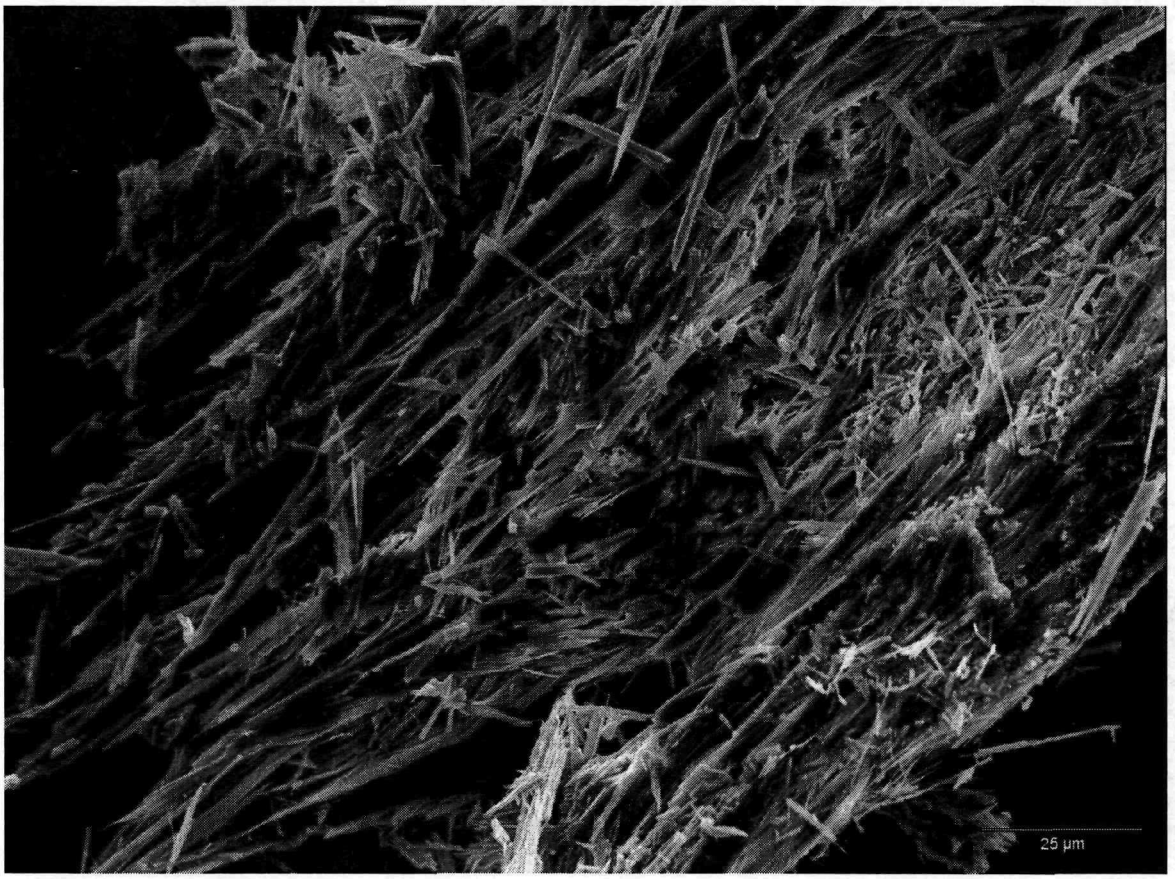
Individual bundles of Libby Amphibole were picked from soil samples at the ESAT Region 8 Laboratory and prepared for analysis by scanning electron microscopy (SEM). Slide mounts of these bundles were initially prepared in a refractive index liquid and the bundles were examined by PLM. Then the refractive index liquid was evaporated off the slides on a hot plate in a fume hood and the bundles of LA were transferred to a SEM stub. Fibers were selected for SEM analysis that showed examples of the range of LA morphologies that may be encountered in field samples. During SEM analysis, energy dispersive spectrometry (EDS) was performed on these fiber bundles and their EDS spectra were found to be consistent with Libby Amphibole.

The SEM analysis was performed by the United States Geological Survey (USGS). Ten of the photomicrographs taken of the LA bundles by the USGS are provided here as a reference to help laboratories understand the range of morphologies of Libby Amphibole that they may encounter in field samples. All of the following pictures are of bundles that were found in field samples collected from the Libby Asbestos Superfund Site in Montana.



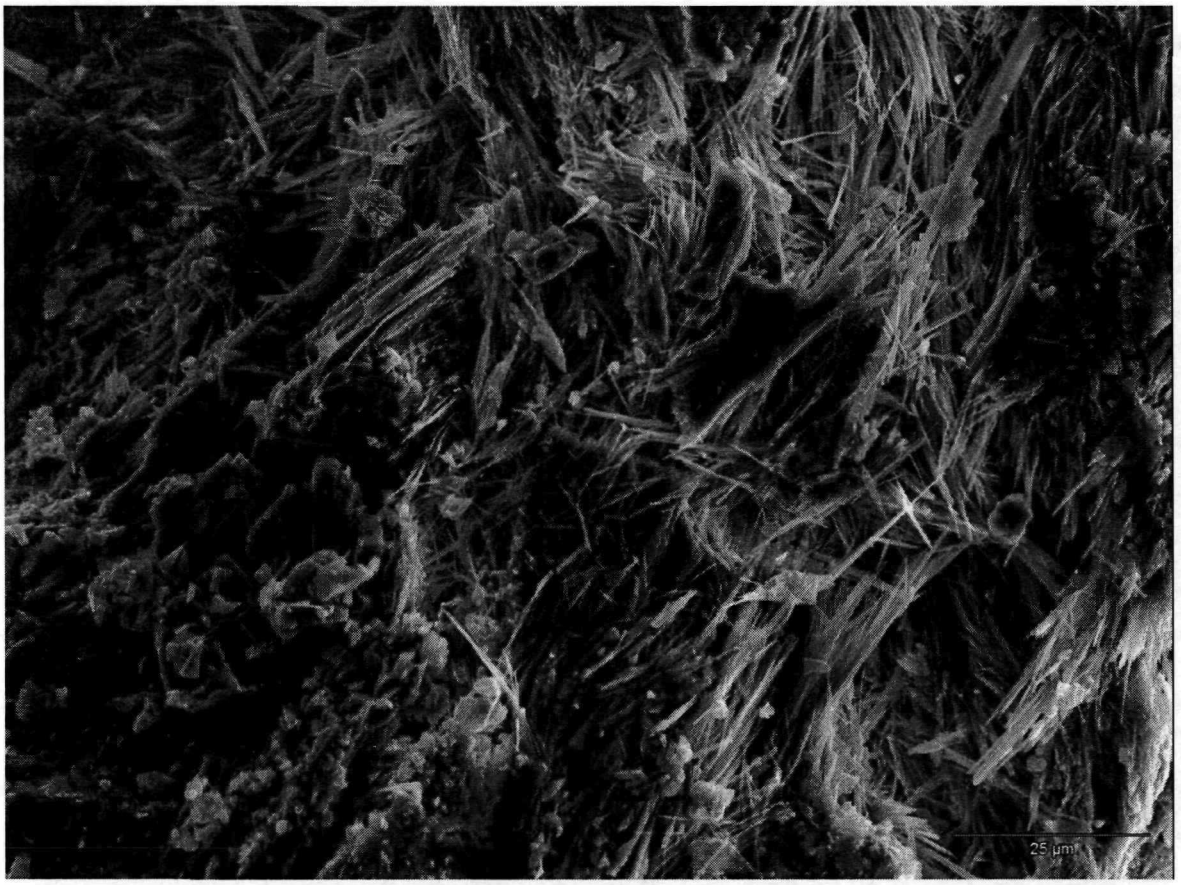
These are typical bundles of Libby Amphibole where the average aspect ratio of the fibers is high and most of the fibers are nearly parallel to one another. Note the scale in microns at the bottom of the photo. These three bundles are all of a size that can be seen with a stereomicroscope and picked out to be placed on a slide for analysis by PLM. The small number "1" at the top of the photo indicates where an EDS spectrum was taken and saved to a file.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.



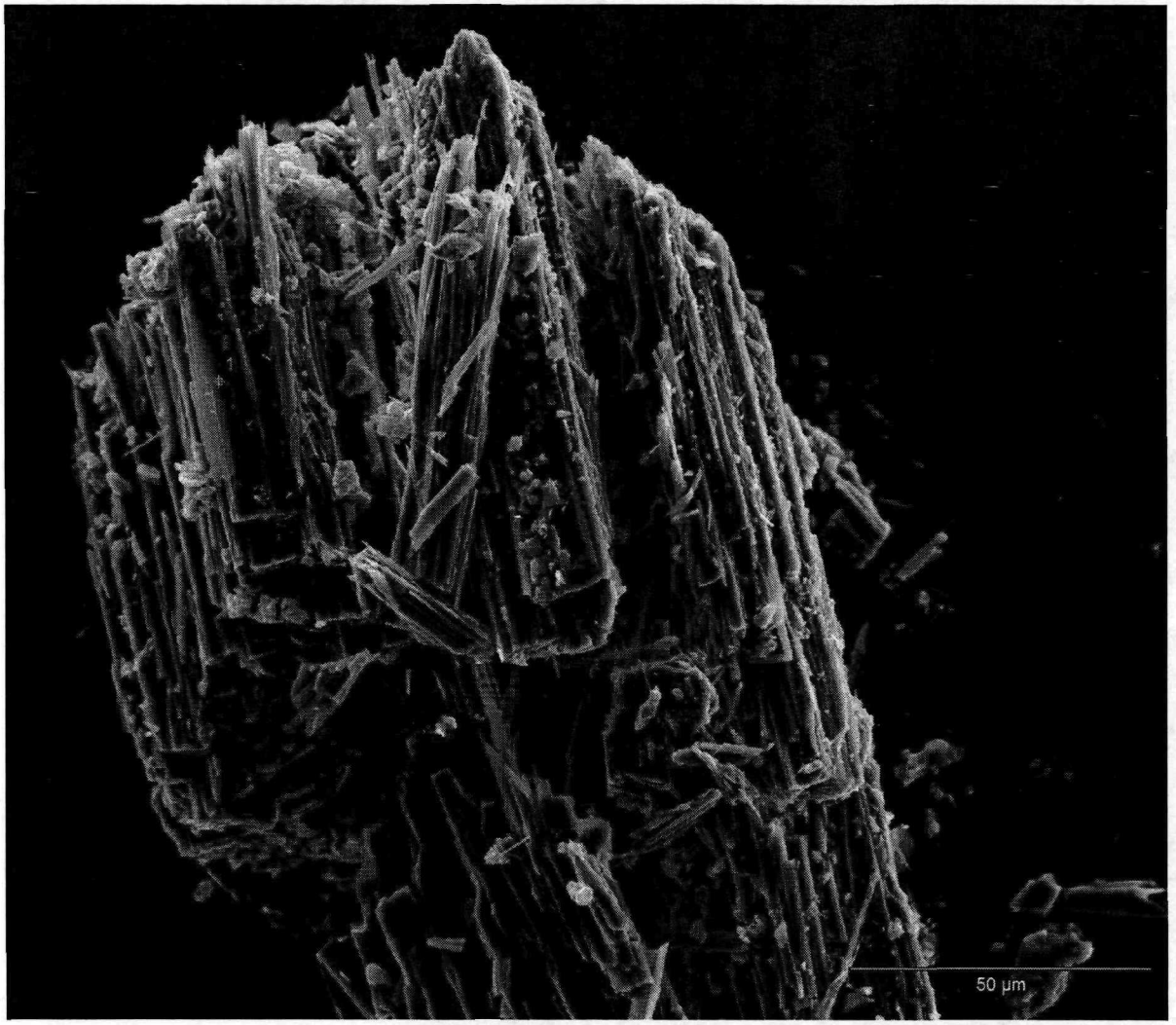
Varying degrees of parallelism can be seen in the fibers that compose bundles of Libby Amphibole. Note that the fibers in this bundle of LA are less parallel than the fibers in the bundles in the previous example.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.



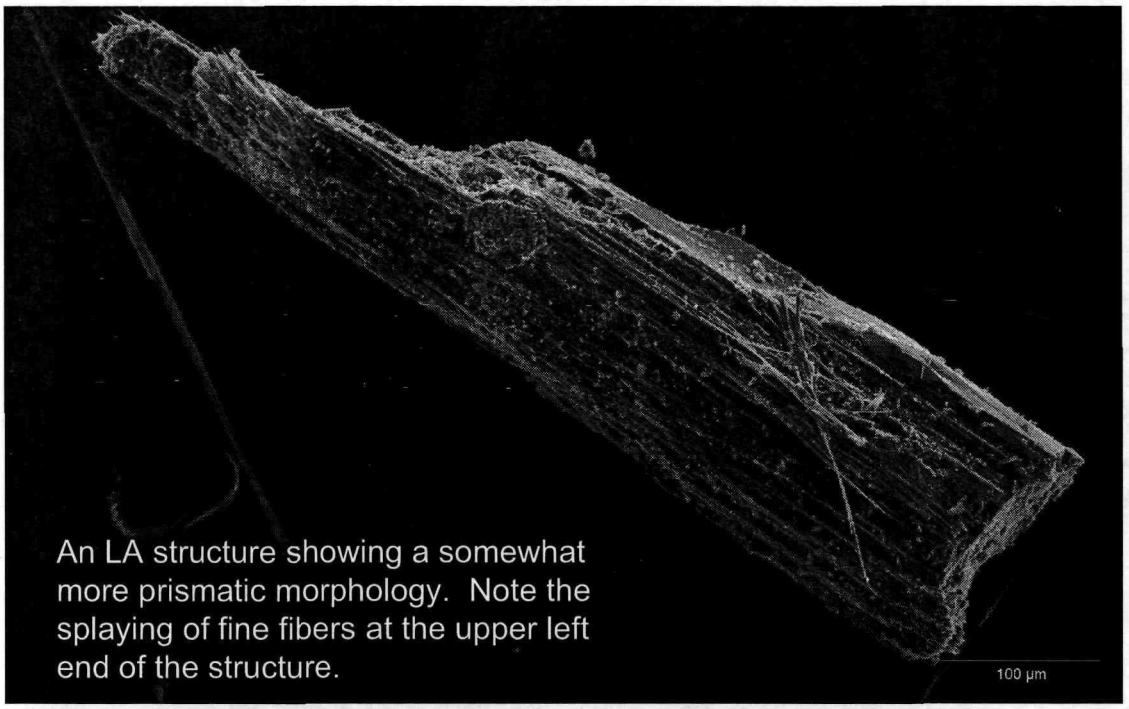
When this bundle of Libby Amphibole was viewed under PLM, its morphology was described as "felted", or "matted", with the fibers crossing at high angles to one another. This is how the bundle appeared when it was subsequently viewed by SEM. The fibrous nature of the "felted" or "matted" morphology is clear at this scale.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

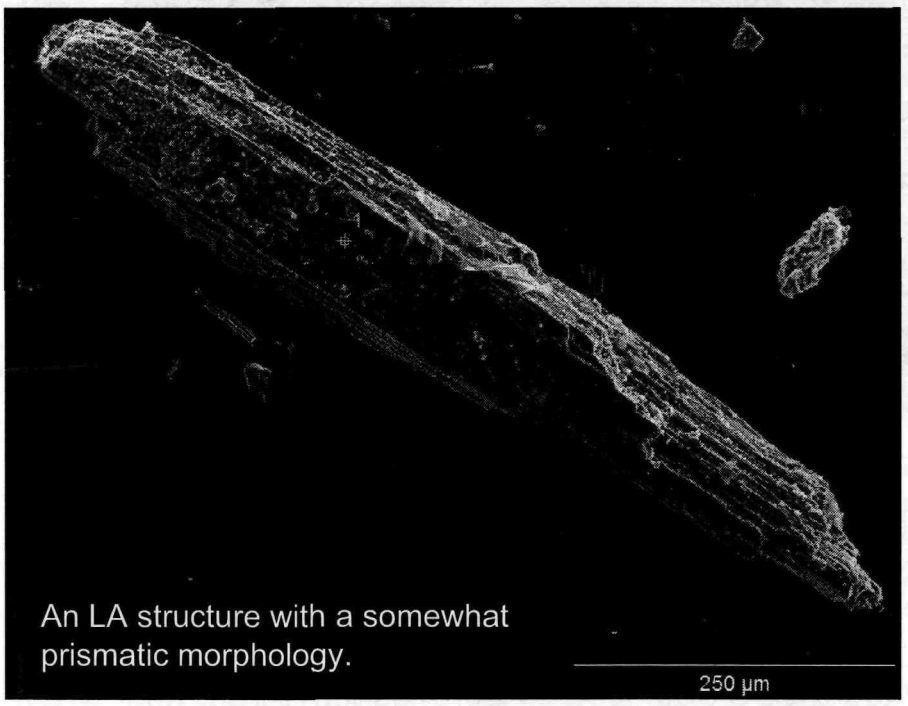


The average aspect ratio of the fibers in this bundle of LA is lower than those of the bundles in the previous examples. However, as seen by SEM, the bundle still splits readily into many small fibers.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

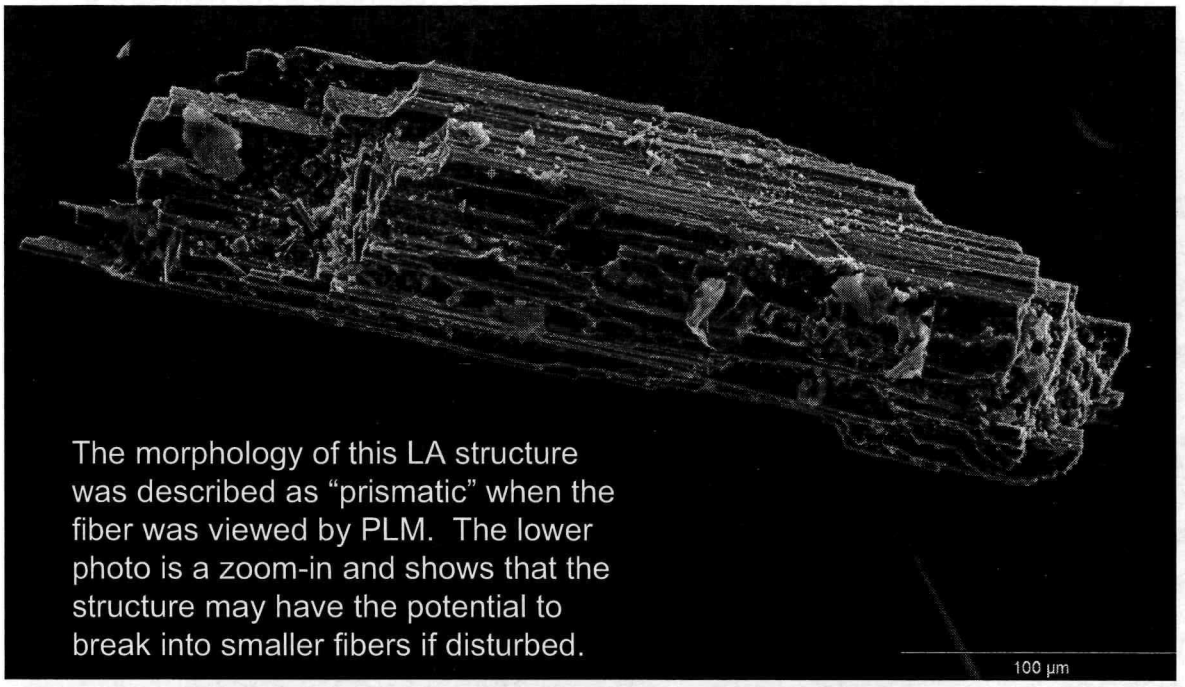


An LA structure showing a somewhat more prismatic morphology. Note the splaying of fine fibers at the upper left end of the structure.

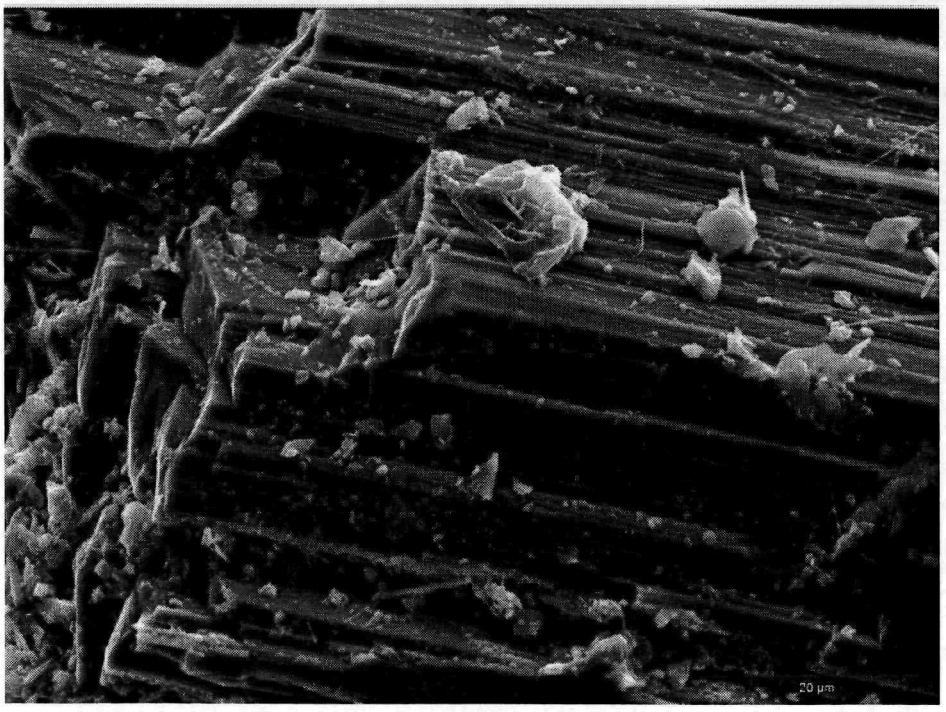


An LA structure with a somewhat prismatic morphology.

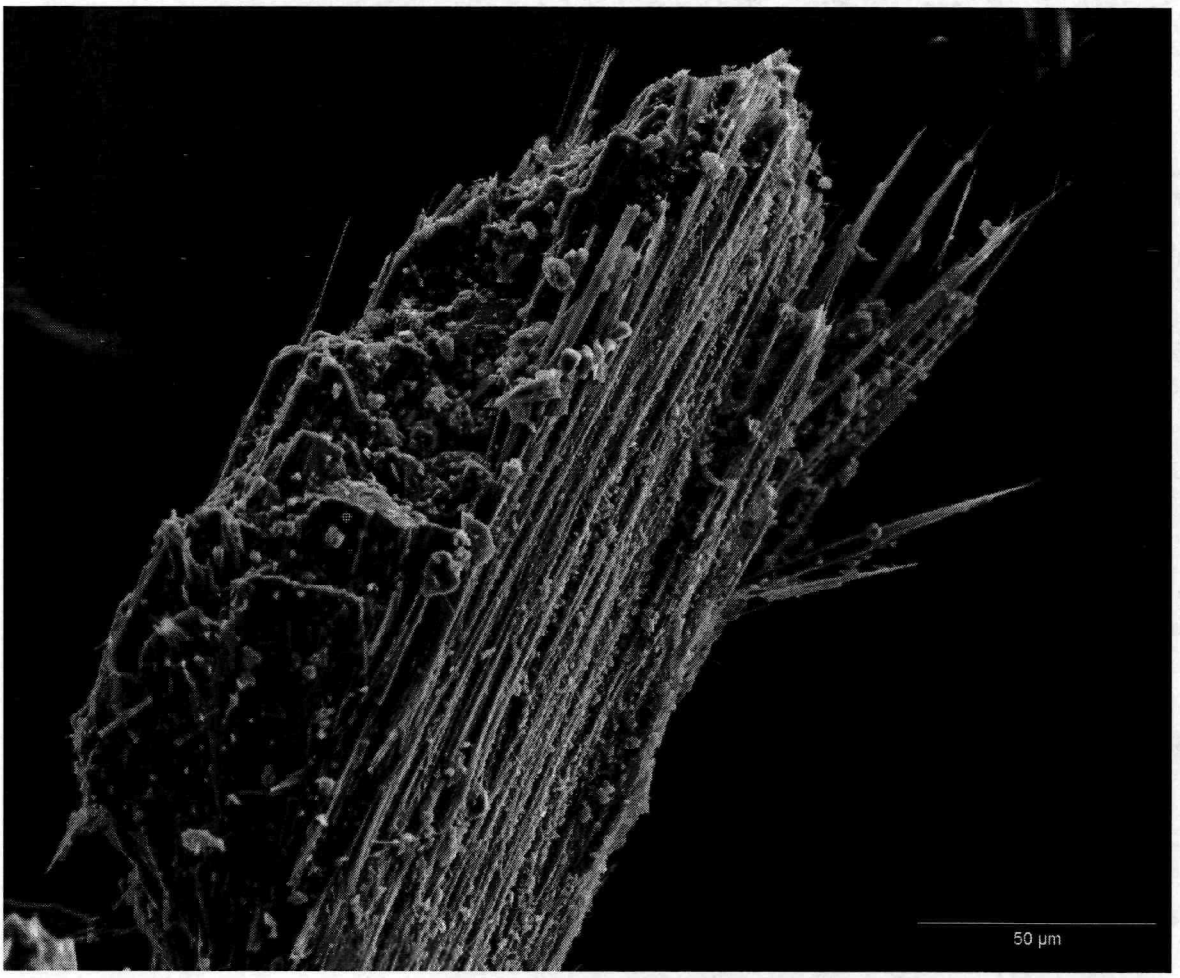
Photographs provided by the USGS and used by permission. Photos for use by the Libby Lab Team only- do not cite or distribute.



The morphology of this LA structure was described as "prismatic" when the fiber was viewed by PLM. The lower photo is a zoom-in and shows that the structure may have the potential to break into smaller fibers if disturbed.

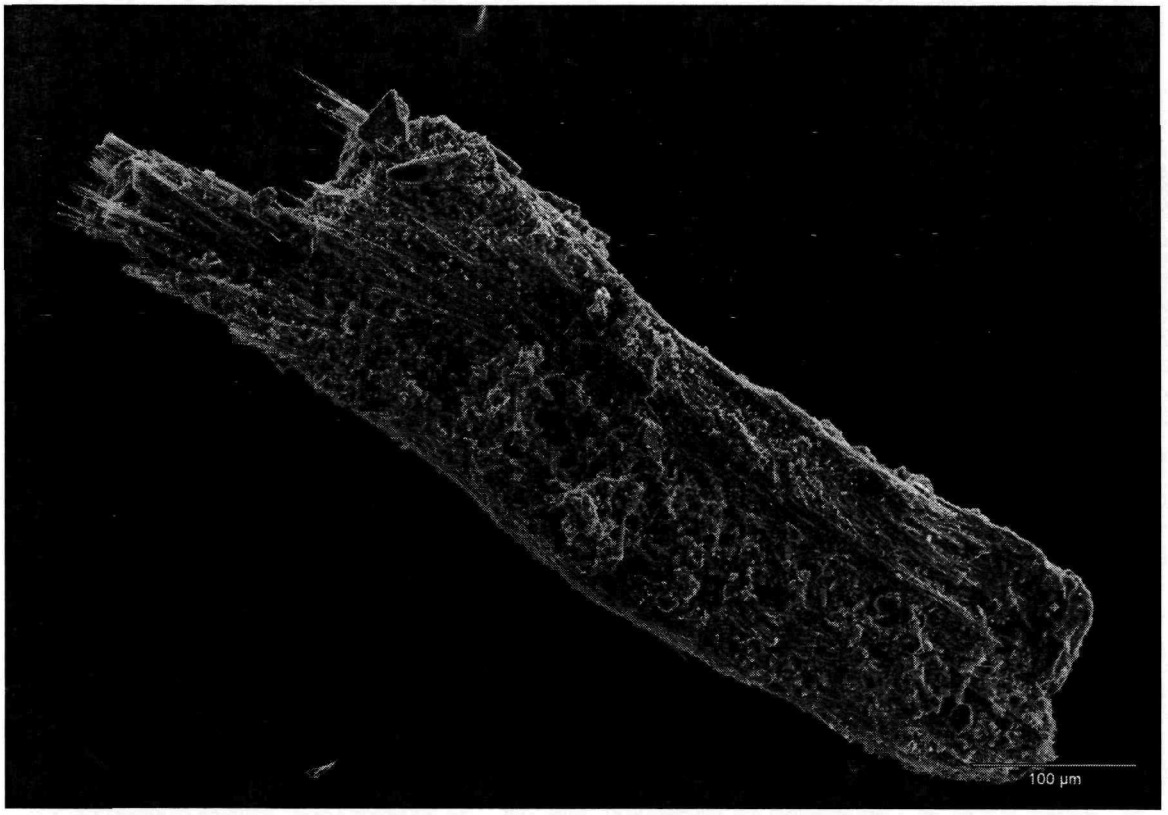


Photographs provided by the USGS and used by permission. Photos for use by the Libby Lab Team only- do not cite or distribute.



This bundle of LA was found either adhered to or grown on a piece of feldspar. Energy dispersive spectrometry (EDS) of the blocky material on the left half of the structure was found to be consistent with potassium feldspar. EDS of the fibrous material on the right, as with all other fiber bundles shown in these photos, was found to be consistent with Libby Amphibole.

Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.



This is a bundle of LA that was found in PLM as either adhered to or grown on a piece of mica. This is how the bundle appeared when it was subsequently viewed by SEM. The EDS spectrum of the platy, rounded material at the lower right end of the structure was found to be consistent with biotite. The EDS spectrum of the fibrous material on the upper left end of the structure was found to be consistent with Libby Amphibole.

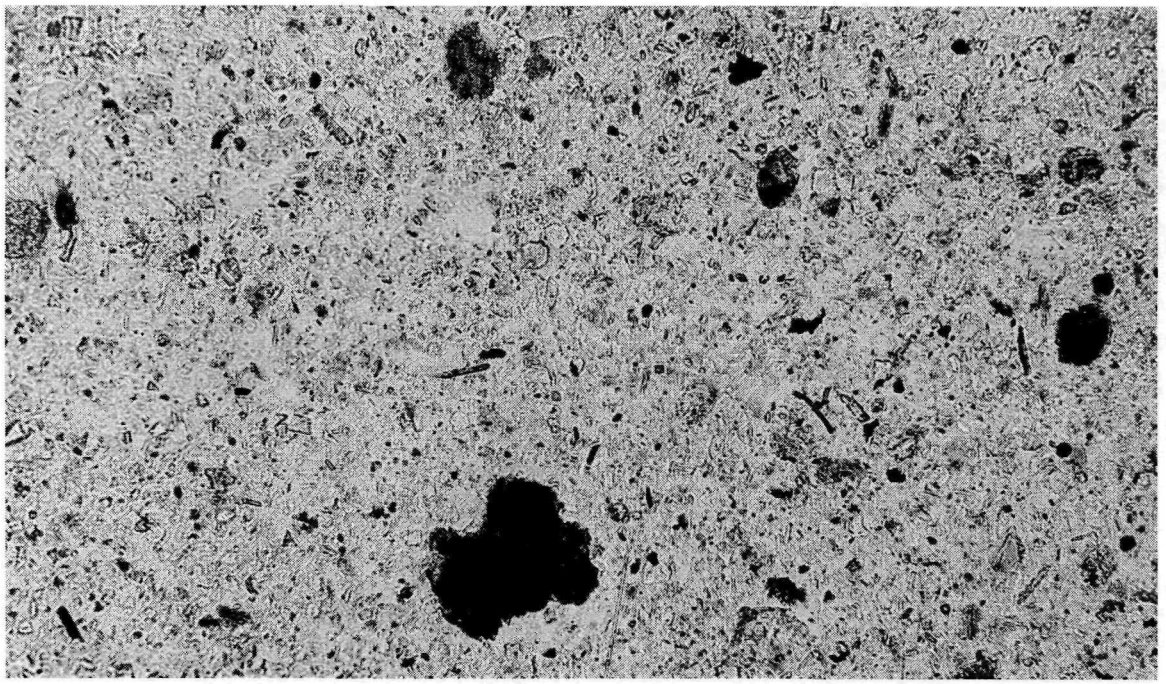
Photograph provided by the USGS and used by permission. Photo for use by the Libby Lab Team only- do not cite or distribute.

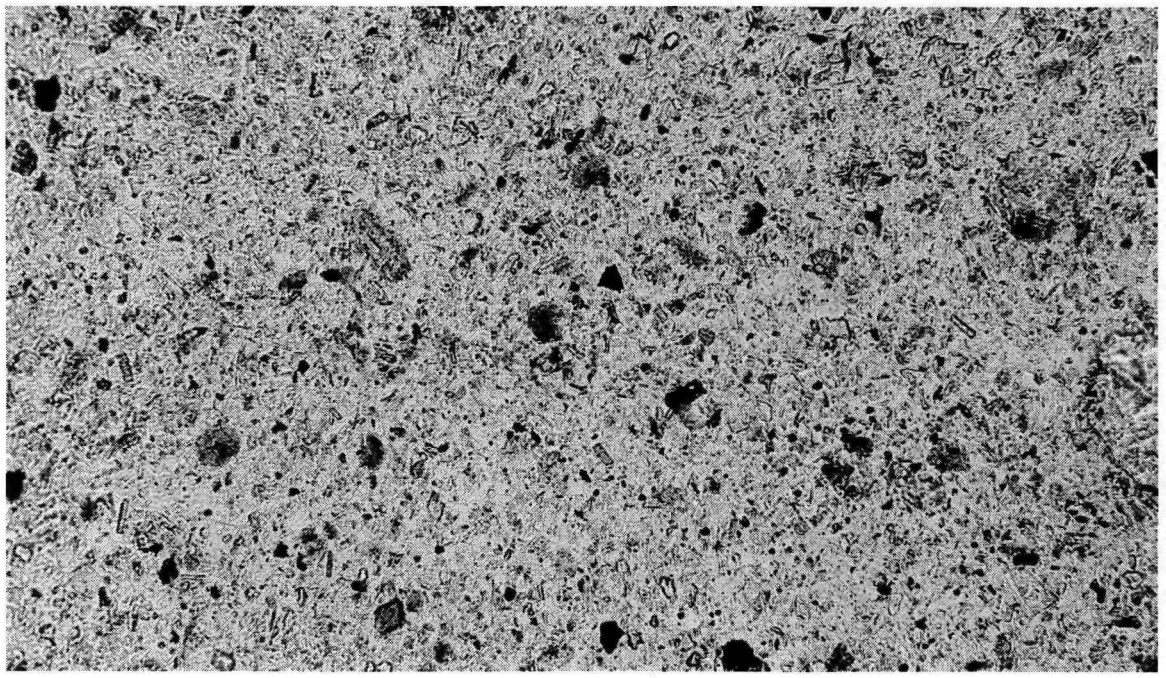
ATTACHMENT 7

Photomicrographs of Representative Fields of View of 0.2% and 1.0% Libby Amphibole Reference Materials

0.2% Libby Amphibole

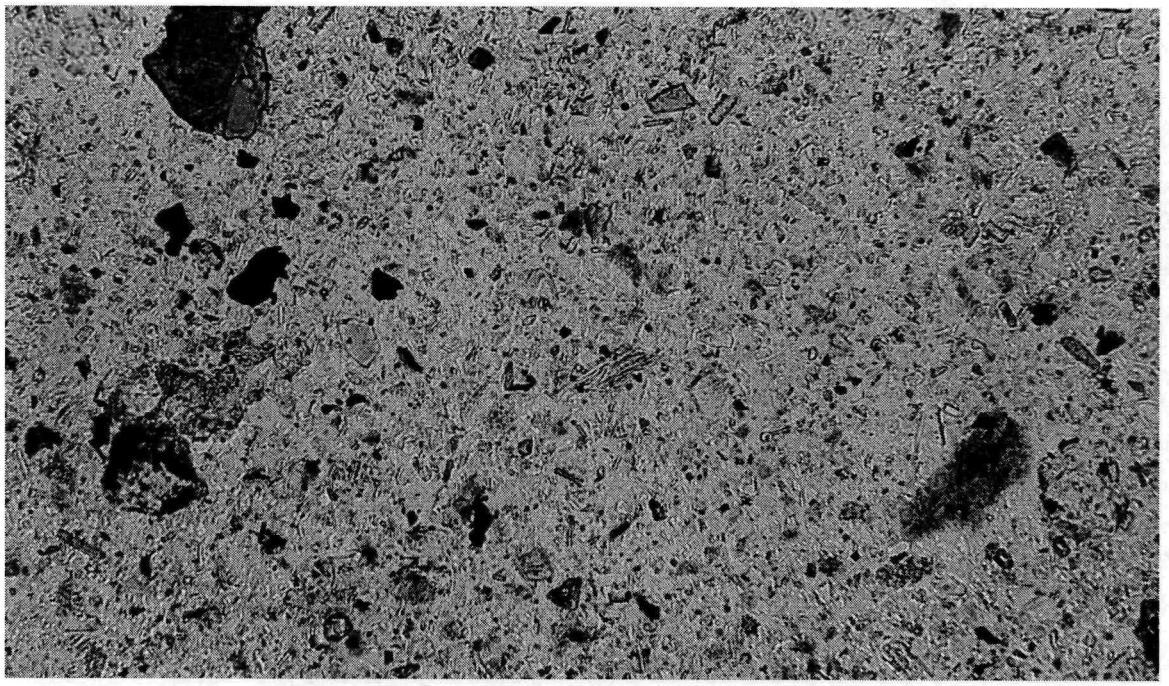
Photomicrographs of representative fields of view of the 0.2% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

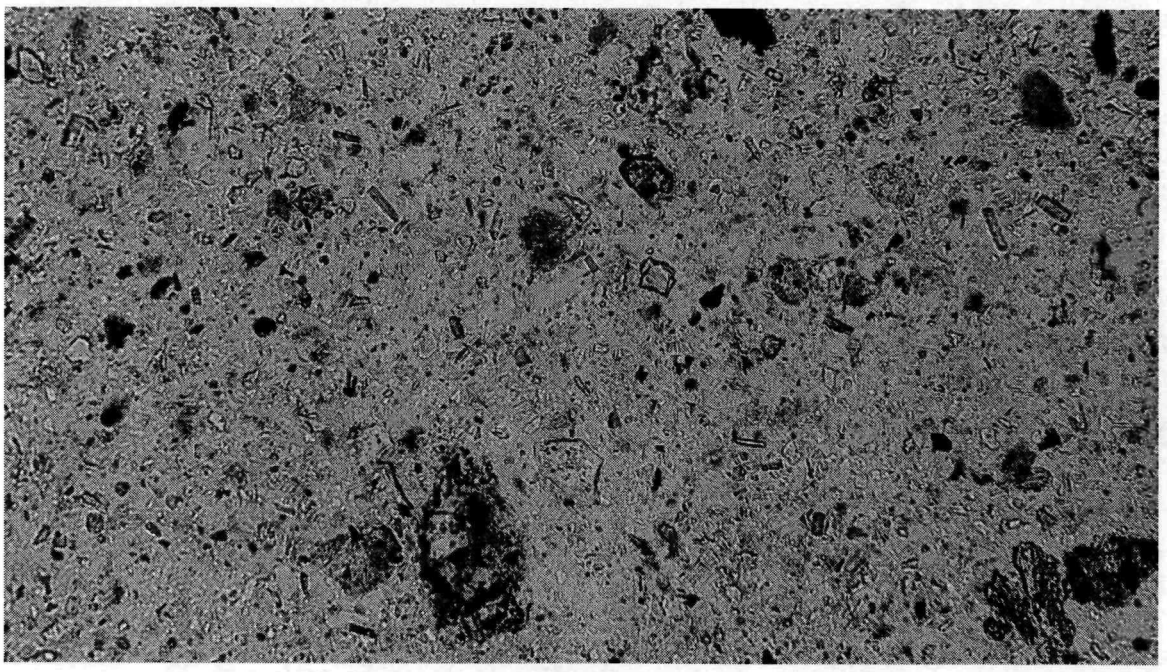




0.2% Libby Amphibole

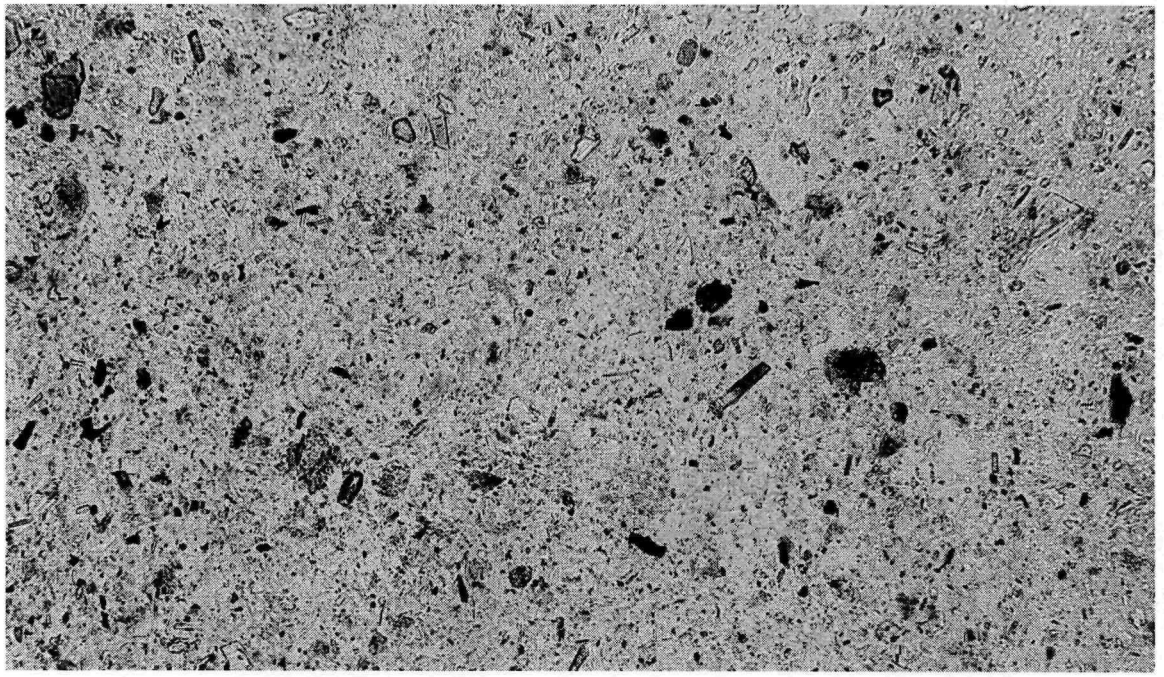
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

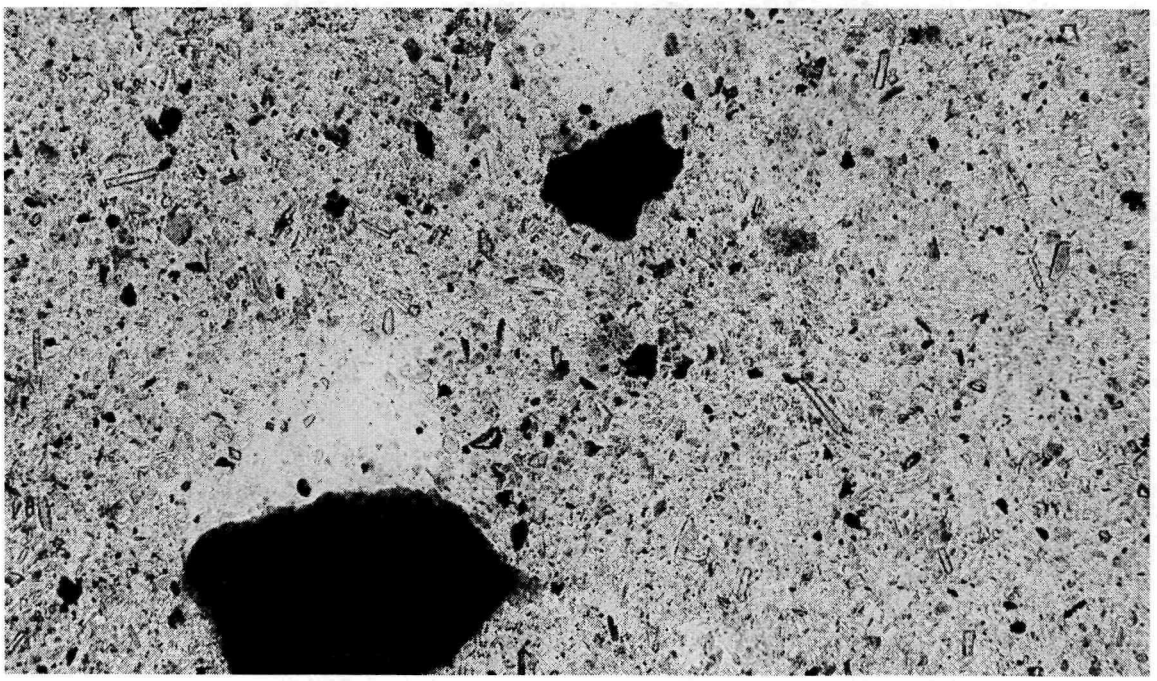




0.2% Libby Amphibole

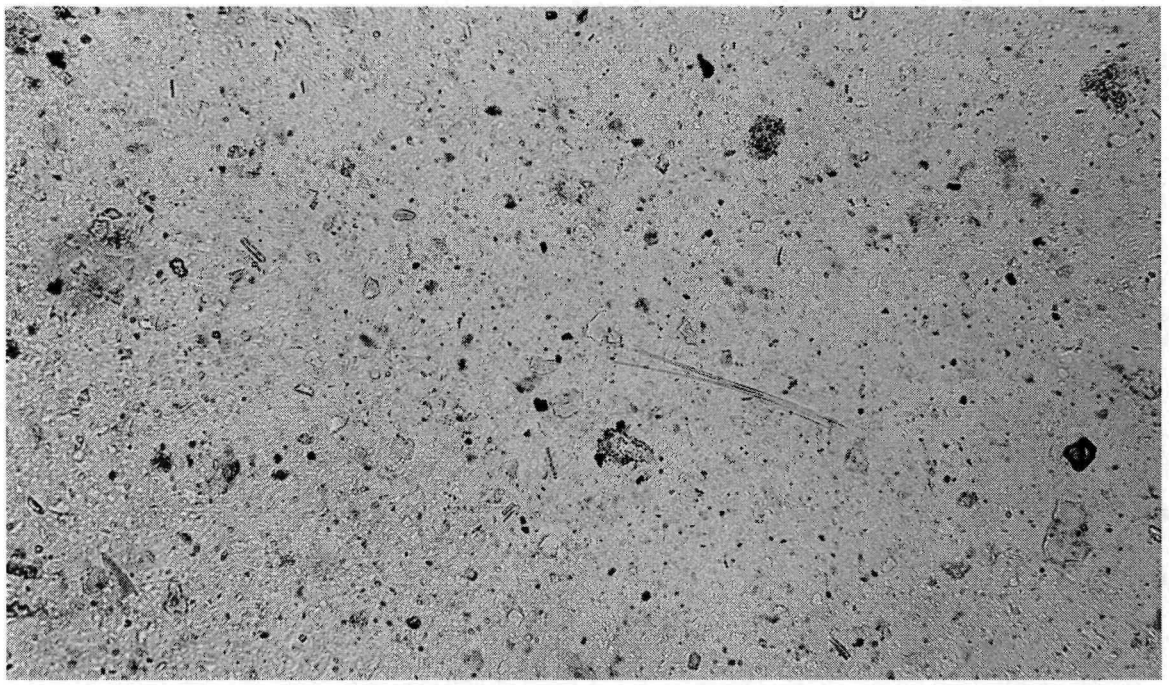
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

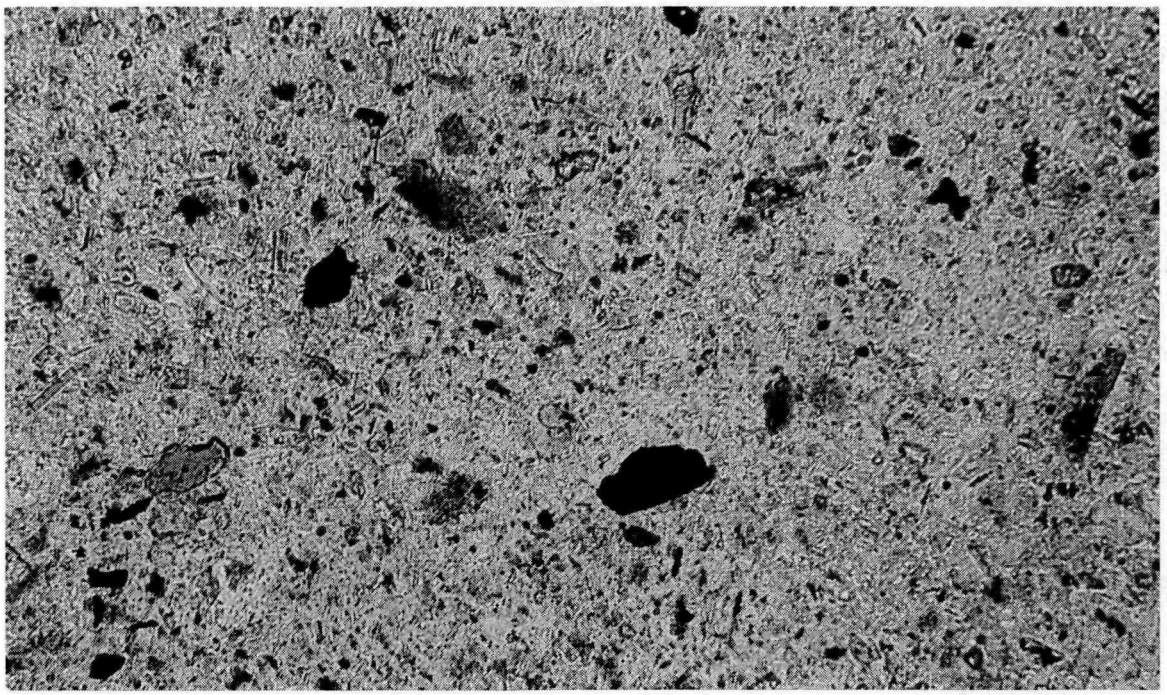




0.2% Libby Amphibole

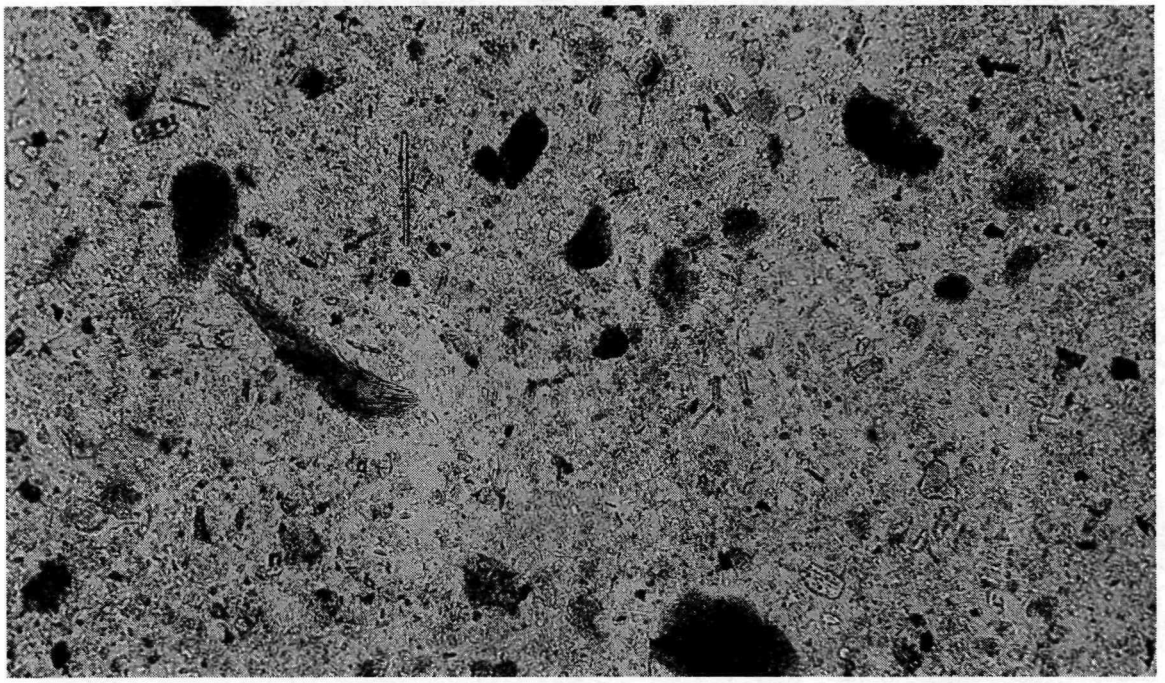
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

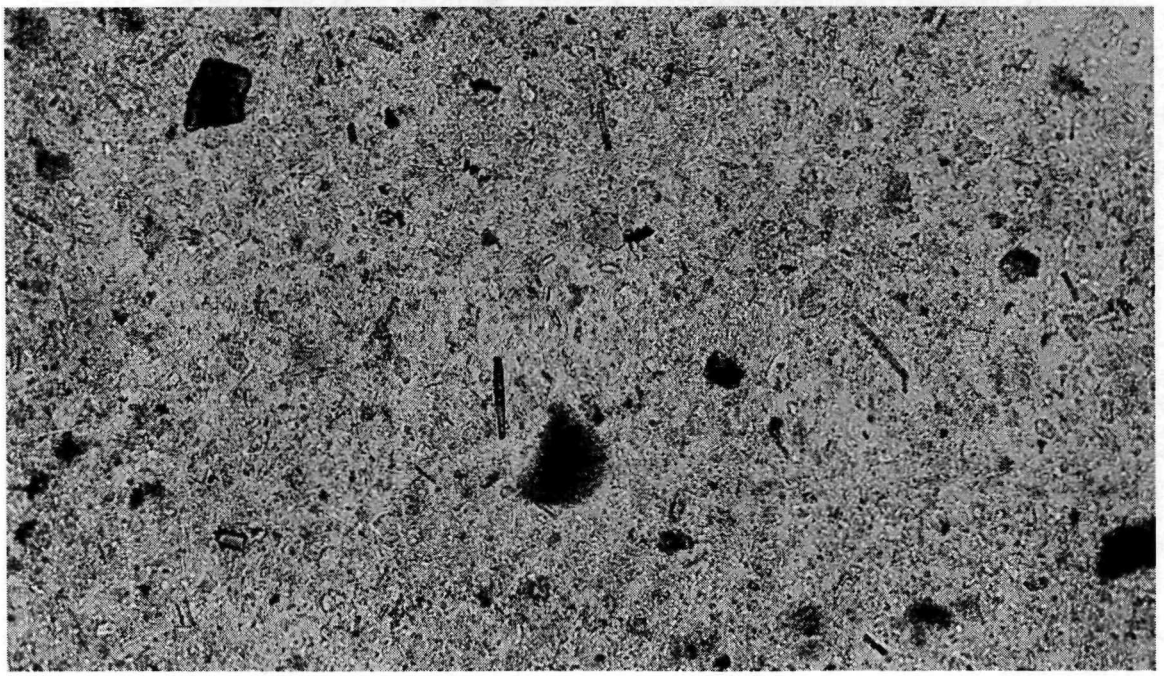




1.0% Libby Amphibole

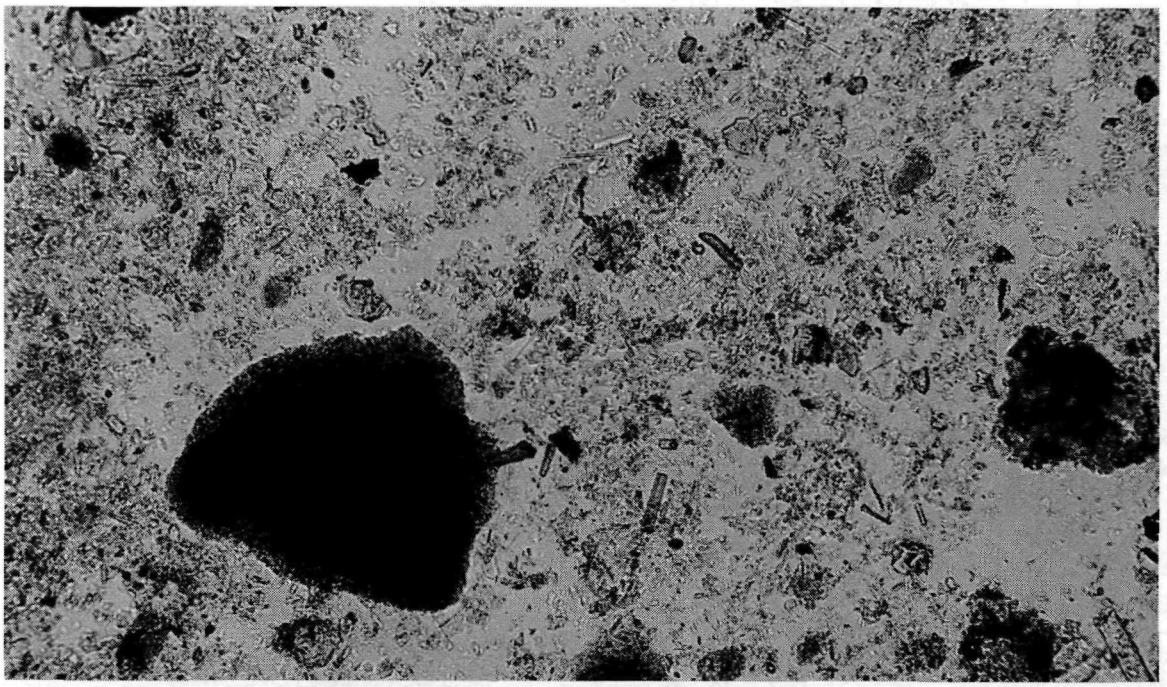
Photomicrographs of representative fields of view of the 1.0% Libby Amphibole by weight Controlled PE Reference Material. All photos taken at 100x, plane light in 1.55 refractive index oil. Width of each picture is approximately 1,500 microns.

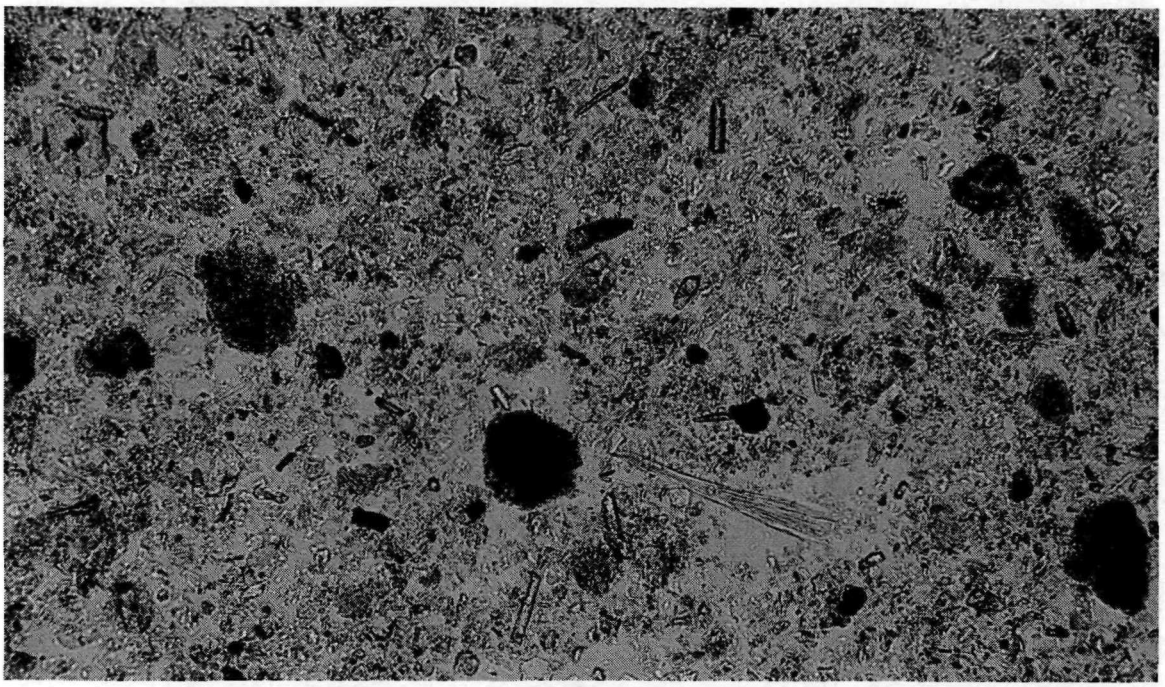




1.0% Libby Amphibole

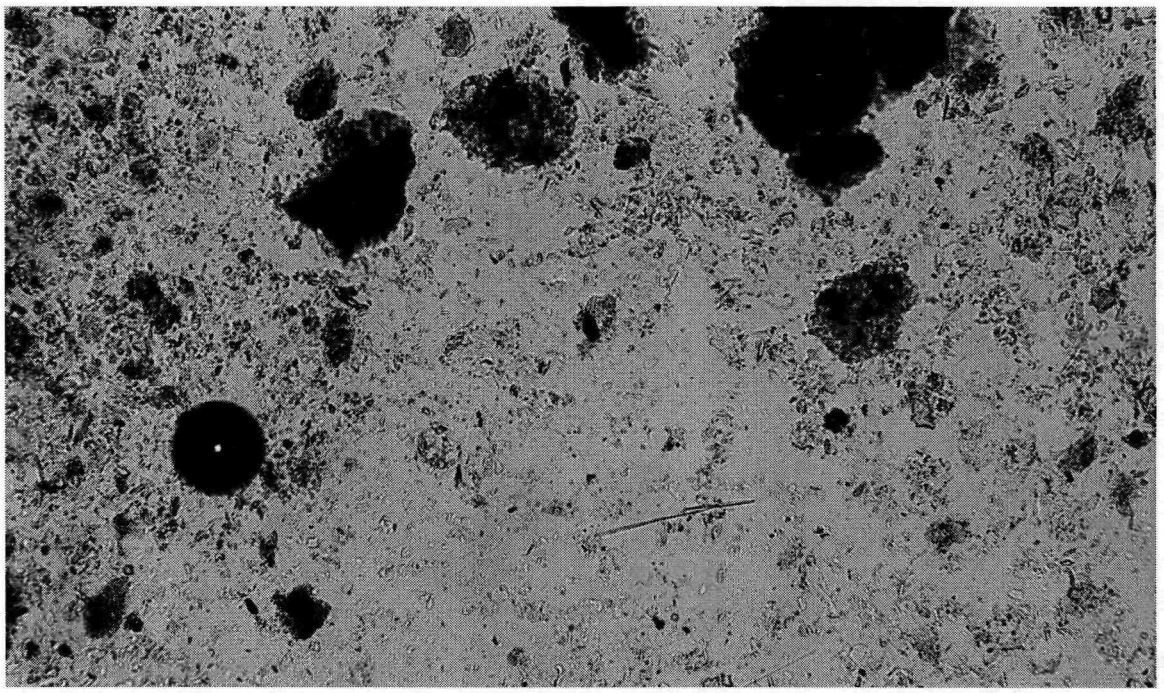
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

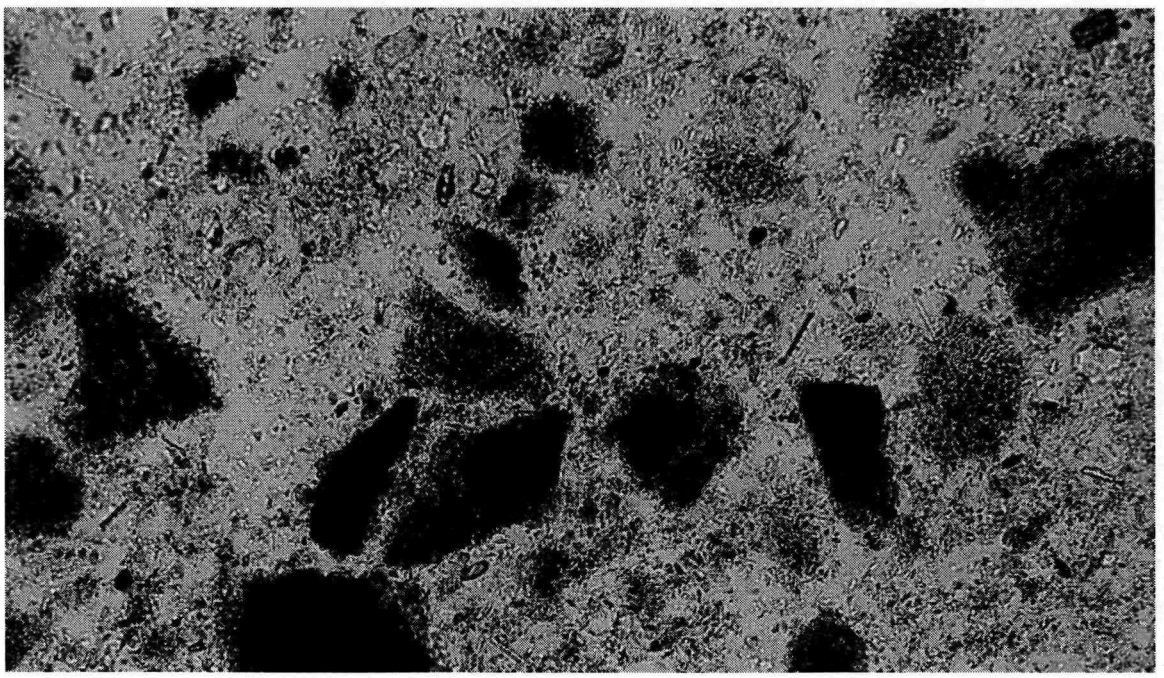




1.0% Libby Amphibole

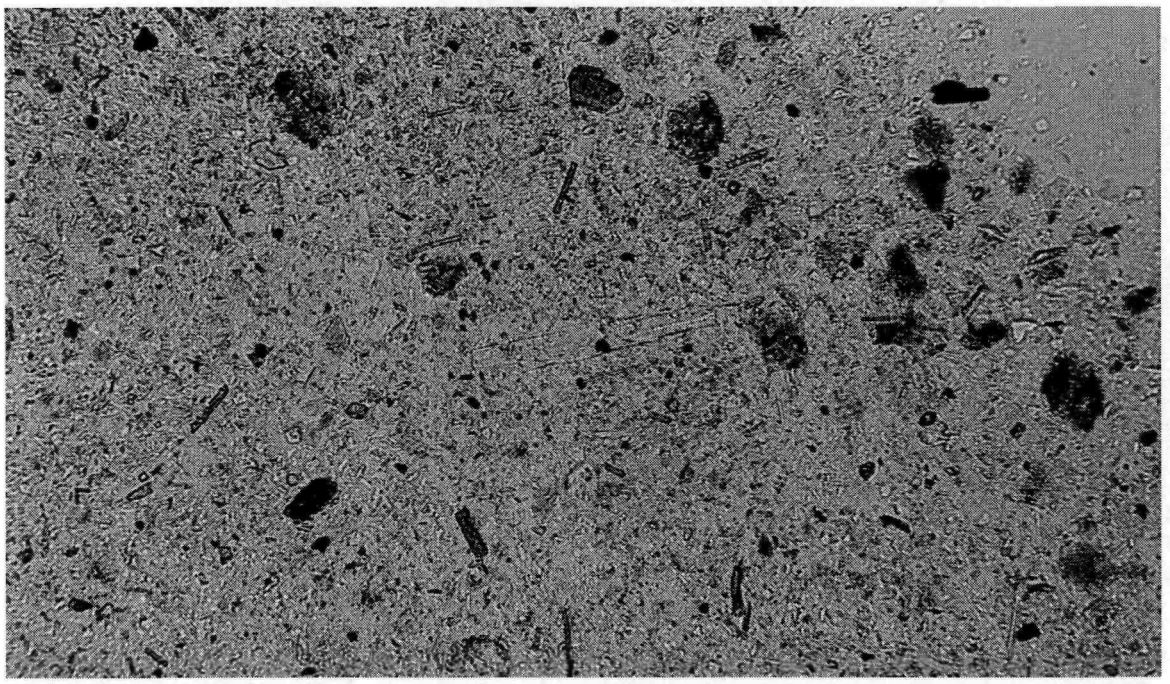
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

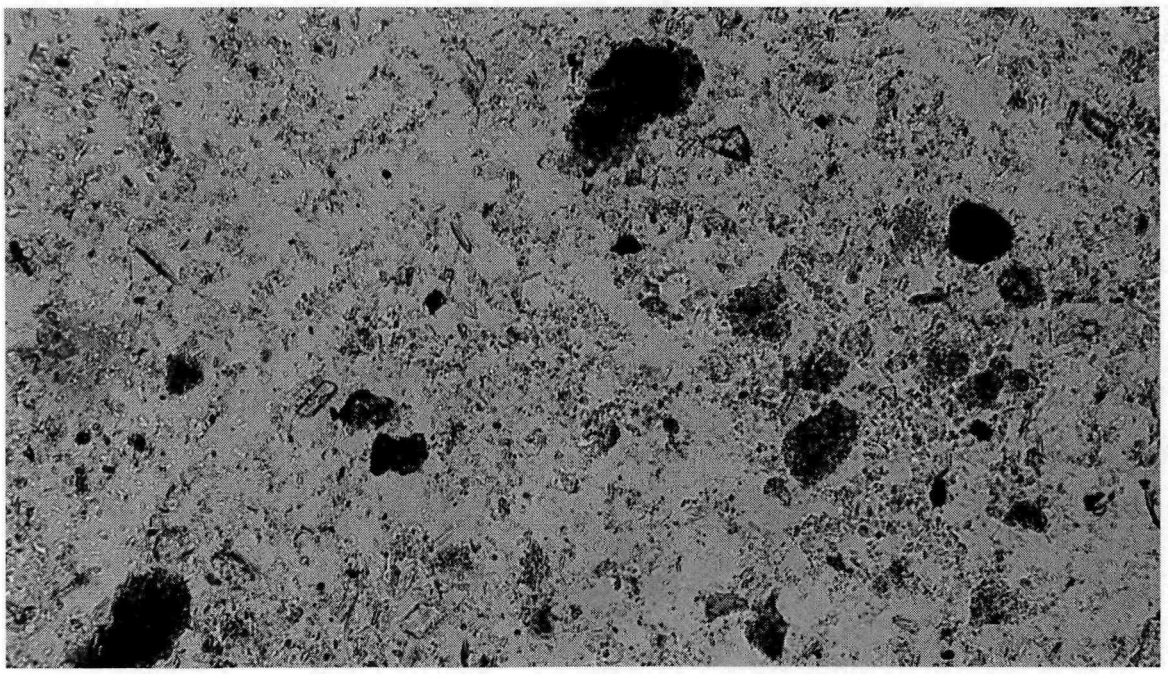




1.0% Libby Amphibole

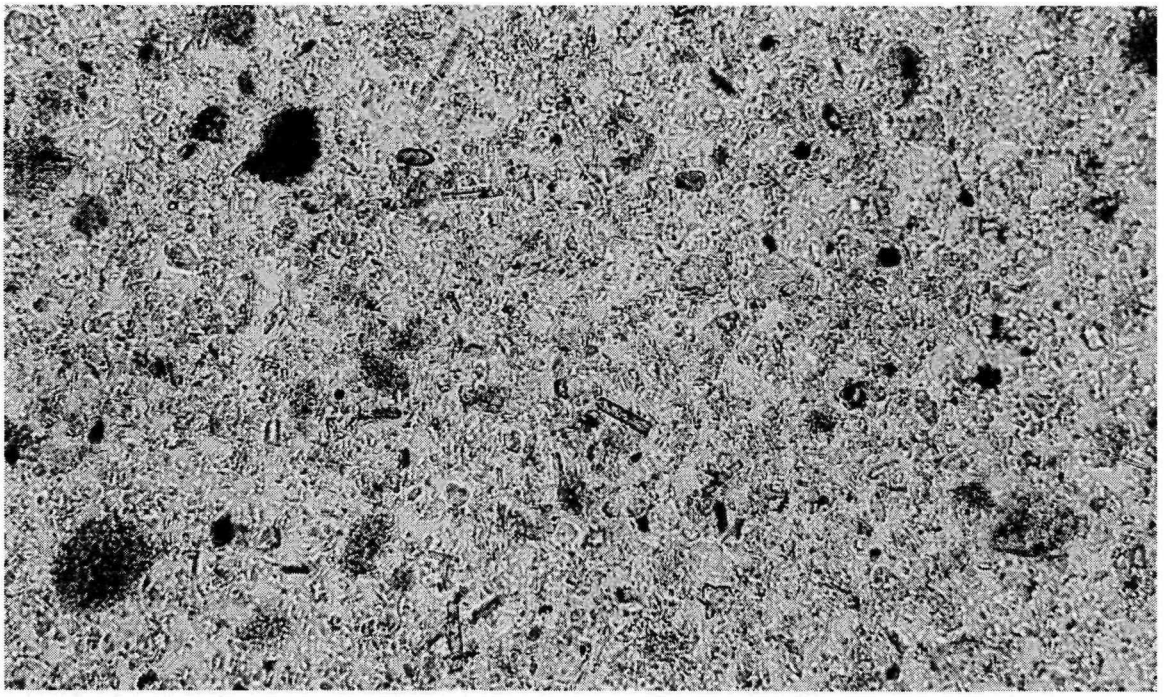
Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

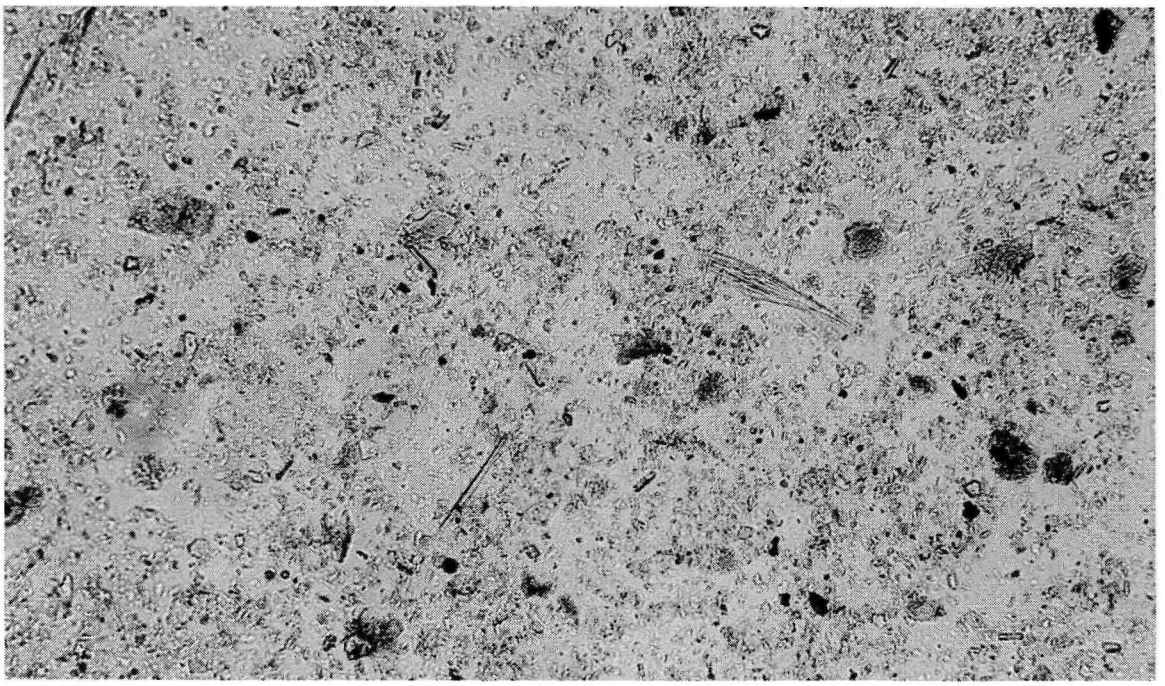




1.0% Libby Amphibole

Photomicrographs of representative fields of view. Width of each picture is approximately 1,500 microns.

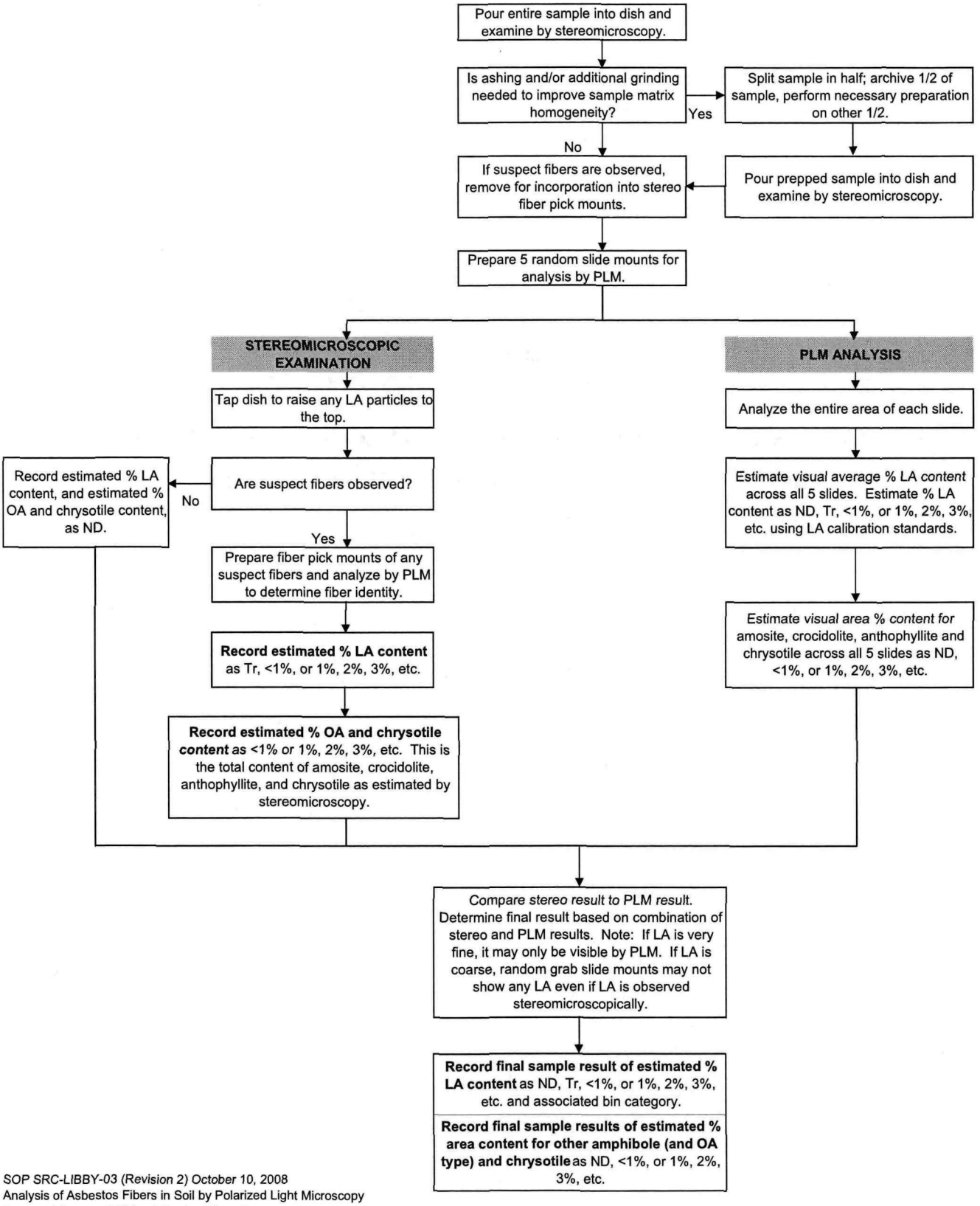




ATTACHMENT 8

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicroscopic Examination and PLM Visual Estimation

Flow Chart for Determining Asbestos Content by Complementary Use of Stereomicrosopic Examination and PLM Visual Estimation



FOR USE AT LIBBY OPERABLE UNIT 3 ONLY

LIBBY OU3 MODIFICATION 1 TO NIOSH 7400 METHOD ANALYSIS OF WATER SAMPLES FOR ASBESTOS BY PCM

Revision 0

Date: May 21, 2009

APPROVALS:

TEAM MEMBER	SIGNATURE/TITLE	DATE
EPA Remedial Project Manager	Bonita Lavelle, USEPA RPM	5/22/09
Modification Author	William Brattin, SRC	5/22/09

Revision	Date	Reason for Revision
0	May 21, 2009	–

1.0 PURPOSE

The purpose of this document is to provide modifications to NIOSH Method 7400 for use at the Libby Superfund Site Operable Unit 3 in the analysis of water samples for Libby Amphibole (LA) by phase contrast microscopy (PCM).

2.0 RESPONSIBILITIES

The Laboratory Director is responsible for ensuring that water samples provided to the laboratory for analysis are prepared and analyzed in accord with the requirements of this modification. It is also the responsibility of the Laboratory Director to communicate the need for any deviations from the modification to the appropriate U.S. Environmental Protection Agency (USEPA) Region 8 Remedial Project Manager or Regional Chemist.

3.0 EQUIPMENT

Sample Preparation

- Sonication device
- Oxygen tank
- Ozone generator
- Plastic and glass tubing

Sample Filtration

- NVLAP-compliant High Efficiency Particulate Air (HEPA) hood
- Particle-free water
- Forceps
- Disposable 47 mm filter funnels
- Side arm filter flask
- Mixed Cellulose Ester (MCE) filters, 47 mm diameter, 0.2 µm and 5.0 µm pore size
- Storage container for filters

Slide Preparation and Analysis by PCM

All equipment required for preparation of slides and filter analysis by PCM analysis is detailed in NIOSH Method 7400.

4.0 MODIFICATION SUMMARY

Samples of water from field sampling or laboratory-based studies are transmitted to a qualified laboratory for analysis of asbestos. At the laboratory, aliquots of water are filtered, and the filters are analyzed by PCM in accord with NIOSH Method 7400 as specified in the applicable Sampling and Analysis Plan. All results are expressed in units of million fibers per liter (MFL).

The results of a PCM analysis should not be interpreted as a reliable measure of the true concentration of asbestos fibers in the water. This can only be provided by TEM analysis. Rather, the primary utility of analysis by PCM is speed (and low cost). The results of PCM analyses are intended to provide a relative measure of concentration, in order to judge, in real time, whether concentration values are changing over time in an unexpected way.

5.0 SAMPLE PREPARATION

The project-specific Sampling and Analysis Plans should specify if and how water samples should be prepared for analysis. In some cases, no preparation is needed other than ensuring the sample is well-mixed before filtration. In other cases, it may be appropriate to use sonication to disperse clumps of fibers that may be present, or to use sonication and ozone treatment combined, as detailed in EPA Method 100.1 Step 6.2, especially in samples where microbial growth may be present.

6.0 FILTER PREPARATION

After sample preparation (if needed), one or more aliquots of water from each sample will be filtered through 47 mm MCE filters with 0.2 μm pores, using a backing filter with pore size of 5 μm. The volume of water filtered should be selected to provide a filter loading of about 100-1000 asbestos structures per mm^2 on the filter.

For water samples in which it is possible to estimate the concentration before analysis (e.g., samples from a laboratory-based toxicity test), the appropriate volume may be estimated as follows:

$$\text{Volume (mL)} = \frac{\text{Target Loading (s/mm}^2\text{)} \cdot \text{Effective Filter Area (mm}^2\text{)}}{\text{Expected Concentration (s/mL)}}$$

For example, assuming an effective filter area of 1295 mm^2, for the analysis of a sample with an expected concentration of 100 MFL (1E+05 s/mL), a loading of about 500 s/mm^2 would be expected after filtration of about 6 mL.

For water samples for which the concentration can not be reasonably estimated before analysis (e.g., most field samples), then it may be necessary to prepare a series of filters, each with a different volume of water. Typically, this will be done by filtering aliquots of 100 mL, 30 mL, and 10 mL of the sample. Select the filter from the dilution series yielding the largest possible application volume which does not result in an overloaded sample (> 2000 structures per mm^2). If the 10 mL aliquot is overloaded, the laboratory shall prepare a dilution of the sample by removing 5 mL of the remaining volume and diluting to 100 mL. From this secondary dilution, prepare a second series of filters using 60 mL, 20 mL, and 6 mL (corresponding to 3.0 mL, 1.0 ml, and 0.3 mL of the original suspension).

7.0 PCM ANALYSIS

All water samples submitted for analysis by PCM will be analyzed in basic accord NIOSH Method 7400.

Slide Preparation

Remove a wedge of about ¼ of the sample filter. Prepare one slide for PCM examination as described in steps 7-9 of NIOSH 7400. Archive the remaining filter for potential use in TEM examination (see below) or for potential re-preparation of additional PCM slides.

Counting Rules

PCM counting rules are specified in Step 18 and Appendix B of NIOSH 7400. In brief, record all structures that are longer than 3 μm, have an aspect ratio (length:width) of 3:1 or higher, and have approximately parallel sides.

Stopping Rules

Stopping rules for PCM analysis should be specified in the project-specific Sampling and Analysis Plan (SAP). In the absence of explicit stopping rules, the PCM analysis should continue until at 20 fields of view (FOVs) have been examined. After this, continue until either a) 100 structures have been recorded, or b) 200 FOVs have been examined.

Data Recording and Electronic Data Deliverable

The total number of structures observed and total number of FOVs examined should be recorded on the most recent version of the Libby site-specific laboratory bench sheets and electronic data deliverable (EDD) spreadsheet ("PCM Water EDD.xls").

8.0 QUALITY CONTROL

The project-specific Sampling and Analysis Plan should specify the types and number of laboratory quality control (QC) samples that should be prepared during the project. In the absence of information in the sampling and Analysis Plan, default guidelines for QC samples are provided in Table 1. This table includes default requirements on the frequency that these QC analyses should be performed, how samples will be selected for QC analyses, the acceptance criteria and corrective actions for these analyses. It is the responsibility of the laboratory manager to ensure that QC requirements are met.

9.0 REFERENCES

National Institute for Occupational Safety and Health (NIOSH). 1994. Asbestos and Other Fibers by PCM. NIOSH 7400 (Issue 2). August 15,1994.

TABLE 1
LABORATORY QUALITY CONTROL SAMPLE DEFAULT REQUIREMENTS [a]

Lab QC Type & Description	Analysis Frequency [b]	Acceptance Criteria	Corrective Action(s)
Lab Blank A filter that is prepared using laboratory water.	1% (1 per 100 analyses)	Structure loading rate on the filter is less than 7 f/mm^2 in an analysis of 100 FOVs.	The laboratory shall immediately investigate the source of the contamination and take steps to eliminate the source of contamination before analysis of any investigative samples may continue.
Repreparation Prepared by applying a second aliquot of sample water to a new filter, which is then prepared and analyzed in the same fashion as the original filter.	2% (1 per 50 analyses) See note [c]	No more than 5% of the original-repreparation pairs are statistically different from each other at the 90% confidence interval. See note [d]	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, etc).
Blind Recounts A slide that has been analyzed is re-labeled and re-submitted (blind) for a second analysis within the same laboratory.	2% (1 per 50 analyses) See note [c]	No more than 5% of the original-recount pairs are statistically different from each other at the 90% confidence interval. See note [d]	A senior laboratory analyst shall determine the basis of the discordant results, and take appropriate corrective action (e.g., re-training in sample and slide preparation, counting rules, etc).

[a] Unless specified otherwise in the project-specific sampling and analysis plan or quality assurance project plan.
[b] When calculating the number of QC analyses required for a project, round up to the nearest whole number.
[c] To be selected by the laboratory in accord with the procedures in Attachment 1 in Libby Laboratory Modification LB-000029.
[d] See Attachment 4 in Libby Laboratory Modification LB-000029 for details on performing this statistical comparison.

Request for Modification
to
Laboratory Activities
LB-000029b

Instructions to Requester: E-mail form to contacts at bottom of form for review and approval.
File approved copy with Data Manager (CDM). Data Manager distributes approved forms as follows:
All Labs Applicable forms – copies to: EPA, Volpe, CDM, All project labs
Individual Labs Applicable forms – copies to: EPA, Volpe, CDM, Initiating Lab

Method (circle one/those applicable): [TEM-AHERA] [TEM-ISO 10312] PCM-NIOSH 7400 NIOSH 9002
EPA/600/R-93/116 [ASTM D5755] EPA/540/2-90/005a SRC-LIBBY-03
Other: _____

Requester: Lynn Woodbury Title: Technical consultant
Company: Syracuse Research Corporation Date: December 7, 2006

Description of Modification:
Permanent clarifications to laboratory-based Quality Control (QC) sample analysis. The purpose of the attached is to standardize the frequency of analysis and procedures for interpretation of the results for laboratory-based Quality Control (QC) samples for TEM analyses of air and dust. The general concepts presented in this modification may also be used for soil and water, but specific details regarding the frequency and interpretation of laboratory QC samples will need to be adjusted for these media.

Reason for Modification:
This modification is needed to standardize the frequency with which different types of QC samples are prepared in different laboratories in the program, and to ensure that all results are evaluated in accord with a standard set of criteria.

Potential Implications of this Modification:
There are no potential negative implications resulting from this standardization of QC procedures.

Laboratory Applicability (circle one): [All] Individual(s) _____

Duration of Modification (circle one):
Temporary Date(s): _____
Analytical Batch ID: _____
Temporary Modification Forms – Attach legible copies of approved form w/ all associated raw data packages

[Permanent] (Complete Proposed Modification Section) Effective Date: _____
Permanent Modification Forms – Maintain legible copies of approved form in a binder that can be accessed by analysts.

Data Quality Indicator (circle one) – Please reference definitions on reverse side for direction on selecting data quality indicators:

[Not Applicable] **Reject** **Low Bias** **Estimate** **High Bias** **No Bias**

Proposed Modification to Method (attach additional sheets if necessary: state section and page numbers of Method when applicable): _____

Technical Review: _____ Date: _____
(Laboratory Manager or designate)

Project Review and Approval: _____ Date: 4/25/07
(Volpe Project Technical Leader or designate)

Approved By: _____ Date: 4/25/07
(USEPA Project Chemist or designate)

DATA QUALITY INDICATOR DEFINITIONS

Reject - Samples associated with this modification form are not useable. The conditions outlined in the modification form adversely effect the associated sample to such a degree that the data are not reliable.

Low Bias - Samples associated with this modification form are useable, but results are likely to be biased low. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated low.

Estimate - Samples associated with this modification form are useable, but results should be considered approximations. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimates.

High Bias - Samples associated with this modification form are useable, but results are likely to be biased high. The conditions outlined in the modification form suggest that associated sample data are reliable, but estimated high.

No Bias - Samples associated with this modification form are useable as reported. The conditions outlined in the modification form suggest that associated sample data are reliable as reported.

QC Sample Type Definitions

There are three categories of TEM laboratory QC samples: Blanks, Recounts, and Repreparations.

Blanks

Lab Blank (LB) – This is a TEM grid that is prepared from a new, unused filter by the laboratory and is analyzed using the same procedure as used for field samples.

Recounts

Recount Same (RS) – This is a TEM grid that is re-examined within the same laboratory and by the same microscopist who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Same TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Recount Different (RD) – This is a TEM grid that is re-examined within the same laboratory but by a different microscopist than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Recount Different TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Interlab (IL) - This is a TEM grid that is re-examined by a microscopist from a different laboratory than who performed the initial examination. The microscopist examines the same grid openings as were counted in the original examination. Interlab TEM analyses for air and dust will be selected in accord with the procedure presented in Attachment 2.

Verified Analysis (VA) – This is a recount of a TEM grid (same grid openings) performed in accord with the protocol for verified analysis as provided in NIST (1994) (provided as Attachment 3). Verified TEM analyses will be selected in accord with the procedure presented in Attachment 1.

Repreparations

Repreparation (RP) – This is a TEM grid that is prepared from a new portion of the same filter that was used to prepare the original grid. Typically this is done within the same laboratory as did the original analysis, but a different laboratory may also prepare grids from a new piece of filter. Repreparations will be selected in accord with the procedure presented in Attachment 1.

Frequency

The minimum frequency for laboratory-based QC samples for TEM analyses (all media combined) shall be as follows:

QC Sample Type	Min. Frequency
Lab blank	4%
Recount same	1%
Recount different	2.5%
Verified analysis	1%
Repreparation	1%
Interlab	0.5%
Total	**10%**

Each laboratory should prepare and analyze lab blank, recount (same, different and verified), and repreparation samples at the minimum frequency specified in the table above. The selection procedure and laboratory SOP for the selection of samples for the purposes of recounts and repreparation are provided in Attachment 1. Samples for interlab comparisons will be selected by EPA's technical consultant (SRC) in accord with the selection procedure and laboratory SOP provided in Attachment 2.

Procedure for Evaluating QC Samples and Responses to Exceptions

The procedure for evaluating QC sample results varies depending on sample type. These procedures are presented below.

Note: The procedures for evaluating QC samples presented below are based in part on professional judgement and experience at the site to date. These procedures and rules for interpretation may be revised as more data are collected.

Lab Blanks.

There shall be no asbestos structure of any type detected in an analysis of 10 grid openings on any lab blank. If one or more asbestos structures are detected, the laboratory shall immediately investigate the source of the contamination and take immediate steps to eliminate the source of contamination before analysis of any investigative samples may begin.

Recounts.

All recount samples (same, different, verified, and interlab) will be evaluated by comparing the raw data sheets prepared by each analyst. Note that the raw data for samples must include sketches for both the initial and QC reanalysis, as described in modification LB-000030. All structure enumeration and measurements will adhere to the established project-specific documentation presented in LB-000016A and LB-000031A. The following criteria will be used to identify cases where results for LA structures are concordant (in agreement) or discordant (not in agreement). These LA criteria were established by microscopists experienced in the analysis of Libby amphibole asbestos, and serve as an initial attempt at review criteria developed using their professional experience. As the database continues to grow and we learn more, these criteria may be revisited and revised. Changes to the criteria for LA structures will be accompanied by scientific justification to support the change. Criteria for concordance on non-LA fibers (OA and C) fibers are the same as described in NIST (1994) (provided as Attachment 3).

Measurement parameter	**Concordance Rule**
Number of LA asbestos structures within each grid opening	For grid openings with 10 or fewer structures, counts must match exactly. For grid openings with more than 10 structures, counts must be within 10%.
Asbestos class of structure (LA, OA, C)	Must agree 100% on chrysotile vs. amphibole. For assignment of amphiboles to LA or OA bins, must agree on at least 90% of all amphibole structures.
LA Structure length	For fibers and bundles, must agree within 0.5 um or 10% (whichever is less stringent) For clusters and matrices, must agree within 1 um or 20% (whichever is less stringent)
LA Structure width	For fibers and bundles, must agree within 0.5 um or 20% (whichever is less stringent). For clusters and matrices, there is no quantitative rule for concordance.

Whenever a recount occurs in which there is one or more discordance, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will use the results of the validated analysis to determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc). Whichever analytical result is determined to be correct will be identified with the word "Confirmed" in the sample comment field of the electronic data reporting sheet. In the special case where the original and the reanalysis are both determined to have one or more areas of discordance, a third electronic data report will be prepared that contains the correct results. This will be identified as QA Type = "Reconciliation". The laboratory should maintain records of all cases of discordant results and of actions taken to address any problems, in accord with the usual procedures and requirements of NVLAP. In addition, each laboratory should notify the CDM Laboratory Manager of any significant exceptions and corrective actions through a job-specific (temporary) modification form. The CDM Laboratory Manager will ensure that appropriate Volpe and EPA representatives are notified accordingly.

Repreparations.
Repreparation samples will be evaluated by comparing the total counts for the original and the re-preparation samples. In order to be ranked as concordant, the results must not be statistically different from each other at the 90% confidence interval, tested using the statistical procedure documented in Attachment 4. Whenever an exception is identified, a senior analyst shall determine the basis of the discordant results, and if it is judged to be related to laboratory procedures (as opposed to unavoidable variability in the sample), the laboratory shall then take appropriate corrective action (e.g., re-training in sample and filter preparation, counting rules, quantification of size, identification of types, etc).

Program-Wide Goals

While each laboratory shall monitor the results of the QC samples analyzed within their laboratory and shall take actions as described above, the overall performance of the program shall be monitored by assembling summary statistics on QC samples, combining data within and across laboratories. The program-wide goals shall be interpreted as follows:

QC Sample Type	Metric	Program-Wide Criteria		
		Good	Acceptable	Poor
Lab Blanks	% with ≥1 asbestos structures	0% - 0.1%	0.2% - 0.5%	>0.5%
Recounts	Concordance on LA count	>95%	85-95%	<85%
	Concordance on type (chrysotile vs. amphibole)	>99%	95%-99%	<95%
	Concordance on LA length	>90%	80%-90%	<80%
	Concordance on LA width	>90%	80%-90%	<80%
Repreps	Concordance on LA concentration/loading	>95%	90-95%	<90%

As the database continues to grow and we learn more, these project-wide goals may be revisited and revised. Changes to the project-wide goals will be accompanied by appropriate justification to support the change.

REFERENCES

NIST. 1994. Airborne Asbestos Method: Standard Test method for Verified Analysis of Asbestos by Transmission Electron Microscopy – Version 2.0. National Institute of Standards and Technology, Washington DC. NISTIR 5351. March 1994.

ATTACHMENT 1

Selection Procedure and Laboratory SOP for Recounts (RS, RD, VA) and Repreparations (RP)

Selection Procedure

As specified in the Frequency section above, the frequency of Recount Same (RS) should be 1%, the frequency of Recount Different (RD) should be 2.5%, the frequency of Verified Analyses (VA) should be 1%, and the frequency of Repreparations (RP) should be 1%, corresponding to a total within-laboratory QC frequency of 5.5% for these analysis types. This is approximately 1 QC sample per 20 field samples. Based on this frequency, it is possible to determine which laboratory job(s) will have one or more samples selected for recount analysis or repreparation.

For those laboratory jobs in which a recount or repreparation sample is to be selected, the analyst should record the total number of structures observed in each sample. The sample(s) selected for recount or repreparation should be those within the laboratory job with the highest number of structures per grid opening (GO) area examined (calculated as the number of GOs evaluated * the GO area). When selecting samples for repreparation, if possible, preferentially select samples in which the total number of GOs is 40 or less. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the selection of samples with 40 GOs or less will reduce analytical costs associated with repreparations. When selecting samples for recount, it is not necessary to impose a minimum or maximum number of GOs because concordance is evaluated on a GO and structure basis, rather than a concentration basis. If all samples within the laboratory job are non-detect, a non-detect sample may be selected. A non-detect sample should be preferentially selected, every 10th selection.

This selection procedure will ensure that the recount analyses and repreparations yield a dataset best suited to assess concordance[1].

Laboratory SOP for Recount Analyses

1. For recount samples, re-analyze the selected sample in accord with the appropriate procedures for each type of recount (RS, RD, or VA). If more than 10 GOs were evaluated in the original analysis, the original analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the recount analysis. The original analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures per GO area examined, which may be analyzed in the event that a selected GO is damaged and cannot be re-evaluated.

2. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Recount Same", "Recount Different", or "Verified Analysis", as appropriate. Be sure that the grid and GO names match exactly with the names evaluated in the original analysis (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the original analyst or laboratory director to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. If there is one or more discordant GOs between the original analysis and the recount analysis, the sample will undergo verified analysis as described by NIST (1994), and the senior laboratory analyst will determine the basis of the discordance, and will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

[1] It should be noted that this selection procedure will tend to result in the preferential selection of samples with the highest air concentration/dust loading values. Thus, summary statistics based on laboratory QC samples may tend to be biased high.

4. Submit the recount TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

Laboratory SOP for Repreparations

1. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

2. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

3. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

4. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

ATTACHMENT 2

Selection Procedure and Laboratory SOP for Interlabs (IL)

Selection Procedure

1. On the 1st of each month, EPA's technical consultant (SRC) will compile a list of all samples for which air and dust TEM results (ISO+AHERA+ASTM) were uploaded into Libby V2 Database in the preceding month (e.g., on November 1st, specify a date range of Oct 1-31, 2005). The Libby V2 Database query will be based on the upload date rather than the analysis date to ensure that analyses with an upload in a different month as the analysis date were not excluded[2].

2. Identify the target number of air and dust interlab samples needed to meet the QC requirements for interlabs specified in the Frequency section above (0.5%). This is accomplished by multiplying the desired interlab frequency (0.5%) by the total number of air and dust analyses performed in the preceding month. For example, 178 TEM air analyses in October 2005 * 0.5% = 0.89 (which is rounded up to 1). At a minimum, at least one air and one dust sample will be selected for interlab analysis.

3. For each medium (air and dust), rank order the TEM analyses from the preceding month on the total number of LA structures per GO area examined (calculated as the number of GOs evaluated * the GO area). Selecting from analyses with a high number of LA structures per GO area examined increases the likelihood that the GOs evaluated as part of the interlab analysis will have one or more LA structures.

4. Exclude samples in which the total number of GOs is more than 40 GOs[3]. Exclude any samples that have already been selected for interlab evaluation previously.

5. Select the appropriate number of air and dust interlab samples from the available TEM analyses for which the total number of LA structures per GO area examined is higher than 0 (i.e., LA detects). If the total number of samples with LA detects is equal to the desired number of interlab samples, select all detected samples for interlab analysis. If the total number of samples with LA detects is less than to the desired number of interlab samples, select non-detect samples for interlab analysis. If the total number of samples with LA detects is higher to the desired number of samples, interlab samples will be selected to represent multiple laboratories, selecting those samples with the highest number of LA structures per GO examined first. EPA's technical consultant (SRC) will keep a running total of the number of samples selected by laboratory to ensure that the long-term frequency of interlabs for each laboratory is generally similar.

6. Submit list of selected interlab samples to the CDM Laboratory Manager.

7. Each month, the CDM Laboratory Manager will provide each laboratory with the list of samples selected for Interlab analysis.

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[2] Consider the case where the TEM analysis for sample X-12345 was performed on September 22 and the results were uploaded on October 3. The interlab selection query performed on October 1, if limited to all results analyzed from September 1-30, would not capture the results for X-12345 because they had not yet been uploaded. The interlab selection query performed on November 1, limited to all results analyzed from October 1-31, would also not capture the results for sample X-12345 because the analysis date is outside of the specified range.

[3] Because all interlabs will be reprepared, these interlab repreparation samples will also be evaluated for concordance with the original sample. Because repreparation concordance is evaluated based on concentration, in order to achieve adequate statistical power, repreparations must prepare and evaluate the same number of GOs as the original analysis to achieve a similar sensitivity. Hence, the focusing on samples with 40 GOs or less will reduce analytical costs associated with repreparations.

At the Originating Laboratory:

1. Upon receipt of the interlab sample list from the CDM Laboratory Manager, locate the appropriate sample filter. If less than ¼ of the sample filter is available, contact the CDM Laboratory Manager to identify an interlab replacement sample.

2. Prepare 3 TEM grids using the standard preparation methods for air and dust at the Libby site.

3. Select two grids and read the same number of total GOs as the original analysis, using the TEM counting rules specified by the CDM Laboratory Manager. For example, if 40 GOs were evaluated in the original analysis, read 20 GOs from the first grid and 20 GOs from the second grid during the repreparation. Place the remaining grid in storage.

4. Record the orientation of each grid using the instructions for grid orientation specified in NVLAP (see Attachment 5).

5. When performing the TEM analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

6. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the QC Type as "Repreparation".

7. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

8. Identify which laboratory will perform the interlab analysis in accord with the following table:

Originating Lab	Lab for Interlab Sample #1	Lab for Interlab Sample #2	Lab for Interlab Sample #3	Lab for Interlab Sample #4	Lab for Interlab Sample #5	Lab for Interlab Sample #6...
Hygeia	Batta	MAS	RESI	EMSL-L	EMSL-W	Repeat... (beginning with the Lab identified for Sample #1)
Batta	MAS	RESI	EMSL-L	EMSL-W	Hygeia	
MAS	RESI	EMSL-L	EMSL-W	Hygeia	Batta	
RESI	EMSL-L	EMSL-W	Hygeia	Batta	MAS	
EMSL-L	EMSL-W	Hygeia	Batta	MAS	RESI	
EMSL-W	Hygeia	Batta	MAS	RESI	EMSL-L	

EMSL-L = EMSL, Mobile Lab in Libby
EMSL-W = EMSL, Westmont

9. If more than 10 GOs were evaluated in the repreparation analysis, the repreparation analyst or laboratory director will select the 10 GOs with the highest number of structures to re-analyze in the interlab analysis. The repreparation analyst or laboratory director should also prepare a list of 5 alternate GOs, based on the next 5 GOs with the highest number of structures, which may be analyzed in the event that the selected GO is damaged and cannot be re-evaluated.

10. Ship the grid(s) for the interlab sample to the appropriate laboratory using standard chain of custody procedures. For each interlab sample, include a list of which GOs should be evaluated for each grid. The names of the grid and GOs provided on the chain of custody form should match exactly with those recorded in the original TEM data recording spreadsheet (including dashes, underscores, and spaces).

11. After the interlab laboratory has completed the interlab analysis, it will request copies of the hard copy laboratory benchsheet(s), the grid opening sketches, and TEM file for each interlab sample.

12. If areas of discordance are noted, the senior laboratory analyst from the interlab laboratory will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

At the Interlab Laboratory:

1. For each grid provided for interlab analysis, place the grid into the TEM grid holder ensuring that the grid orientation matches that which was specified by the originating laboratory (see Attachment 5 for details).
2. For the 10 GOs identified for interlab analysis, perform TEM analysis using the analysis method and counting rules specified on the chain of custody. Be sure that the grid and GO names match exactly with the names provided on the chain of custody (including dashes, underscores, and spaces). If a GO cannot be evaluated (e.g., GO is damaged), DO NOT arbitrarily select a different GO for evaluation. Utilize the list of 5 alternative GOs provided by the originating laboratory to select an alternate GO for evaluation. Identify the names of any GOs that could not be evaluated in the comment field along with a brief description of why they could not be analyzed (e.g., grid opening F7 torn, not analyzed).

3. When performing the TEM interlab analysis, identify the relative position of each structure within the grid opening using the template provided as Attachment 6. It is not necessary to sketch the actual structure (as this is already recorded on the hard copy benchsheet), but the analyst should record the structure number which corresponds to the hard copy benchsheet. The analyst should also record the relative position of any non-asbestos mineral (NAM) structures. Use a new template for each grid opening.

4. Record the results using the most recent version of the TEM data recording spreadsheet. Identify the Laboratory QC Type as "Interlab".

5. Submit the TEM spreadsheet to the CDM Laboratory Manager using standard deliverable procedures.

6. Contact the originating laboratory to request copies of the hard copy laboratory benchsheet(s), grid opening sketches, and TEM file for each interlab sample.

7. Perform a verified analysis using the procedures presented in NIST (1994) (provided as Attachment 3).

8. Assess the between-laboratory concordance, both on a GO-by-GO basis and on a structure-by-structure basis, using the Libby-specific recount concordance rules. If areas of discordance are noted, the senior laboratory analyst will contact the originating laboratory to discuss the basis of the discordance. As needed, the senior laboratory analyst will then take appropriate corrective action (e.g., re-training in counting rules, quantification of size, identification of types, etc).

9. Summarize the results of the verified analysis and document any changes in laboratory procedures or analyst training that were implemented to address noted discordances. Provide a copy of this report to EPA Chemist and the CDM Laboratory Manager.

10. Ship the grid(s) back to the originating lab.

ATTACHMENT 3

Airborne Asbestos Method:
Standard Test Method for Verified Analysis of Asbestos
by Transmission Electron Microscopy-Version 2.0.
NIST (1994)

NISTIR 5351

Airborne Asbestos Method: Standard Test Method for Verified Analysis of Asbestos by Transmission Electron Microscopy - Version 2.0

Shirley Turner
Eric B. Steel

U.S. DEPARTMENT OF COMMERCE
Technology Administration
National Institute of Standards
and Technology
Microanalysis Research Group
Surface and Microanalysis Science Division
Chemical Science & Technology Laboratory
Gaithersburg, MD 20899

March 1994

U.S. DEPARTMENT OF COMMERCE
Ronald H. Brown, Secretary

TECHNOLOGY ADMINISTRATION
Mary L. Good, Under Secretary for Technology

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
Arati Prabhakar, Director

Preface

This Interagency Report (IR) is one of a series of IRs that will form the basis of a method for analysis of airborne asbestos by transmission electron microscopy. The form and style of the American Society for Testing and Materials (ASTM) was adopted as a standard format for this series of reports.

1. Scope

1.1 This test method describes a procedure for verified analysis of asbestos by transmission electron microscopy.

1.2 The method is applicable only when sufficient information has been collected during the analyses of a grid square so that individual asbestos structures can be uniquely identified.

1.3 The method is written for the analysis of a grid square by two TEM operators but can be used for more than two operators with slight modifications. Due to the analysis of a grid square by more than one TEM operator, the test method can be applied only when contamination and beam damage of particles are minimized. The two TEM operators can use the same TEM for the analysis or the analyses can be done on different TEMs (in the same or in different laboratories).

1.4 The method can be used with any set of counting rules applied by all analysts. Though the method describes verification of asbestos particles, the method can also be used for verification of analyses of nonasbestos particles if all analysts use the same counting rules.

2. Terminology

2.1 *Definitions:*

2.1.1 *TEM*--transmission electron microscope.

2.1.2 *grid square, grid opening*--an area on a grid used for analysis of asbestos by transmission electron microscopy.

2.1.3 *verified analysis*--a procedure in which a grid opening is independently analyzed for asbestos by two or more TEM operators and in which a comparison and evaluation of the correctness of the analyses are made by a verifying analyst. Detailed information -- including absolute or relative location, a sketch, orientation, size (length, width), morphology, analytical information and identification -- is recorded for each observed structure.

2.1.3.1 *Discussion*--Verified analysis can be used to determine the accuracy of operators and to determine the nature of problems that the analyst may have in performing accurate analyses. Verified counts can be used to train new analysts and to monitor the consistency of analysts over time.

2.2 *Description of Terms Specific to This Standard:*

2.2.1 *counting rules*--rules used to determine the amount of asbestos present in an asbestos- containing sample. Counting rules are a part of most methods for analysis of asbestos by transmission electron microscopy including the AHERA method and the ISO method (see definitions below).

2.2.2 *AHERA method*[1]--procedure for analysis of asbestos by transmission electron microscopy developed by the Environmental Protection Agency with subsequent modifications by the National Institute of Standards and Technology.

2.2.3 *ISO method*[2]--procedure for analysis of asbestos by transmission electron microscopy developed by the International Standards Organization.

2.2.4 *particle*--an isolated collection of material deposited on a grid or filter.

2.2.5 *structure*--a particle or portion of a particle that contains asbestos and that is considered countable under the method used for asbestos analysis. A structure is a basic unit used in many methods of asbestos analysis to report the amount of asbestos present in a particle.

2.2.6 *TEM operator, TEM analyst*--person that analyzes a grid square by transmission electron microscopy to determine the presence of asbestos.

2.2.7 *verifying analyst*--person that compares the analyses of a grid square by two or more TEM operators. The reported asbestos is compared on a structure-by-structure basis by the verifying analyst. Structures that are not matched are relocated and reanalyzed by the verifying analyst. The verifying analyst is

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[1]Code Fed. Reg. 1987, *52* (No. 210), 41826-41905.

[2]ISO 10312 1993, in press.

preferably not one of the TEM operators. If this cannot be avoided, the job of verifying analyst should be rotated between the TEM operators.

2.2.8 *TEM analysis form*--form on which the analysis of a grid square is recorded. The information recorded for a verified analysis should include at least a sketch of the structure and information related to the absolute or relative location, size, identification and analytical data for the reported structures.

2.2.9 *report form*--form on which the evaluation of verified analyses is summarized. The form should be identical to or include all information given in Figure X1.1 of Appendix X1.

2.2.10 *SR (structures reported)*--the number of structures reported by a TEM analyst.

2.2.11 *TP (true positive)*--structure that is: 1) reported by both TEM operators or 2) reported by one operator and confirmed by the verifying analyst, or 3) reported by neither TEM operator but is found by the verifying analyst. The three types of true positives are discussed in the next three terms.

2.2.12 *TPM (true positive-matched)*--structure that is reported on the TEM analysis forms of both TEM operators.

2.2.12.1 *Discussion*--To qualify as a match, the structures should be comparable in the following characteristics: 1) absolute or relative location, 2) appearance in the sketch, 3) orientation, 4) size (length, width), 5) morphology (shape, hollow tube), 6) analytical information (chemistry and/or diffraction data), and 7) identification. In addition, the structures should be reported as countable by both analysts.

2.2.13 *TPU (true postive-unmatched)*--structure that is reported on the TEM analysis form of only one operator and that is confirmed as countable by the verifying analyst.

2.2.14 *TPV (true positive found by verifying analyst)*--structure not found by the two TEM operators but found by the verifying analyst.

2.2.15 *TNS (total number of structures)*--the number of structures determined to be in a grid opening by verified analysis of the grid opening. This value corresponds to the number of unique true positives found by the TEM operators and the verifying analyst.

2.2.15.1 *Discussion*--The value for the total number of structures is not necessarily the actual number on the grid square because both the TEM analysts and the verifying analyst may have missed one or more structures. The probability of a missed structure, however, decreases with an increased number of analysts.

2.2.16 *FN (false negative)*--structure that has not been reported as countable by one of the TEM analysts. False negatives can be divided into two categories-type A and type B as discussed in the next two terms.

2.2.17 *FNA (false negative-type A)*--false negative that was recorded on a TEM analyst's TEM analysis form but not reported as a structure. Some reasons for this type of false negative include: 1) structure misidentified as nonasbestos, 2) confusion with the counting rules, 3) incorrect length determination.

2.2.18 *FNB (false negative-type B)*--false negative that was not recorded on a TEM analyst's TEM analysis form. A reason for this type of false negative is that a structure was missed by an analyst.

2.2.19 *FP (false positive)*--reported particle that is incorrectly identified as a structure. Some reasons for false positives include: 1) structures counted more than one time, 2) materials misidentified as asbestos, 3) confusion with the counting rules, 4) incorrect length determination.

2.2.20 *TN (true negative)*--reported particle that is correctly characterized as zero structures.

2.2.21 *NL (not located structure)*--structure reported on one TEM analyst's TEM analysis form that cannot be located by the verifying analyst.

2.2.21.1 *Discussion*--The value for NL should be zero for most verified analyses, especially if the grid has not been removed from the TEM between the two analysts' counts. If, however, a grid has been removed from an instrument, there is a small possibility of fiber loss.

2.2.22 *AMB (ambiguous structure)*--a structure that 1) is identified as a structure by only one TEM operator and 2) is found by the verifying analyst but cannot be unambiguously identified as a structure due to beam damage, contamination, or other factors.

3. Significance and Use

3.1 The analysis of asbestos by transmission electron microscopy is important for the determination of the cleanliness of air or water and for research purposes. Verified analyses provide more accurate values for the concentration of asbestos on a grid opening than obtained by other methods. The accuracy should increase with an increased number of analysts participating in the verified count.

3.2 The test method can be used as part of a quality assurance program for asbestos analyses and as a training procedure for new analysts. The values for TP/TNS and FP/TNS can be plotted *vs* time on control charts to show improvements or degradations in the quality of the analyses. Experienced analysts should attain TP/TNS values ≥ 0.85 and FP/TNS values ≤ 0.05. The test method can be used to characterize the types and, in many cases, the causes of problems experienced by TEM analysts.

3.3 The average of values obtained for TP/TNS and FP/TNS can be used to determine the analytical uncertainty for routine asbestos analyses.

4. Procedure

NOTE 1-- This test method involves two TEM operators and a verifying analyst. The steps discussed in items 4.1 and 4.2 are to be followed by the person coordinating the analyses by the TEM operators. This person can be one of the TEM operators, the verifying analyst or an independent person (e.g., a quality assurance officer). The steps discussed starting with item 4.3 are to be followed by the verifying analyst.

4.1 Obtain analyses of a grid square for asbestos by two TEM operators. Conduct the analyses independently so that the second operator has no knowledge of the results obtained by the first operator.

4.1.1 Require that the TEM operators record on the TEM analysis form information related to the absolute location of the structures or conduct analyses so that the relative location of the structures can be compared.

NOTE 2– The absolute location of the structures can be recorded by various means including use of a digital voltmeter or computer readable stepping motors to record the position of a structure. To preserve information about the relative location of the reported structures, the analyses must be conducted so that both analysts: 1) orient the grid in the TEM in the same fashion, 2) start the analysis from the same corner of the grid square, 3) initially scan in the same direction, and 4) scan the grid square in parallel traverses.

4.1.2 Require that the TEM operators record on the TEM analysis form a sketch of the structure, the dimensions of the structure, analytical data and whether the structure is countable. The sketch of the structure should include any nearby features that could aid in subsequent identification - for instance, nearby particles, sample preparation features or grid bars.

4.2 Submit the analyses of the two TEM operators to the verifying analyst.

NOTE 3– The remainder of this section describes procedures to be followed by the verifying analyst. The procedure for comparison of the TEM analysis forms is given in items 4.3-4.6 and examples of comparisons of count sheets are given in Figs. X2.1-X2.9 of Appendix 2. Appendix 3 contains a summary of the comparison process (Fig. X3.1) and a flow chart for comparison of structures in the TEM (Fig. X3.2). The procedure for completion of the report form is given in item 4.7.

4.3 Compare the two TEM analysis forms on a structure-by-structure basis. If a match of asbestos structures is observed, label both sketches with a TPM(number) either in the sketch box or in a column specifically designated for verified counts. An example is given in Fig. X2.1 of Appendix X2.

NOTE 4– The next step in the procedure (item 4.4) is optional. The most prudent approach is to examine unmatched structures in the TEM (item 4.5).

4.4 Determine if the status of any of the unmatched structures can be unambiguously decided by examining the TEM analysis forms. If there is ambiguity in determining the status of a structure, the verifying analyst must examine the structure in the TEM as described in items 4.5-4.6. The comparison of TEM analysis forms and labelling of unmatched structures can be relatively straight foward as shown in Fig. X2.2 - X2.4 of Appendix X2 or more complex as described in the next item.

4.4.1 For most cases, the identification of true positives, false positives and false negatives can be done on a structure-by-structure basis. This cannot be done, however, in cases where analysts determine different numbers of countable structures in an asbestos-containing particle. In such cases, both analysts should be assigned one TPM(number) for identifying the particle as containing countable asbestos. The remaining structures are assigned TPU, FP or FN depending on the particular situation. Examples of such cases are given in Fig. X2.5 and Fig. X2.6 of Appendix X2.

4.5 Determine the status of any remaining unlabelled structures by examining the grid square in the TEM. Examples of TEM analysis forms containing structures that must be examined by transmission electron microscopy are given in Figs. X2.7 - X2.9 of Appendix 2. For each unlabelled structure requiring examination by transmission electron microscopy, follow items 4.5.1-4.5.7 and 4.6 until the structure is labelled. If there is another unlabelled structure, go back to item 4.5.1 and repeat the procedure. Continue until all structures are labelled. A summary flow chart for examination by TEM is given in Fig. X3.2. The procedure and flowchart do not cover the counting discrepancy discussed in item 4.4.1. If such a situation is recognized, the verifying analyst should follow the procedure given in item 4.4.1 and in the examples in Figs. X2.5 and X2.6.

NOTE 5-- The procedure in items 4.5.1-4.5.7 should cover the great majority of cases encountered when attempting to determine the status of the structures. There may, however, be more complex situations not covered in the procedure. If so, the verifying analyst should apply the basic principles outlined in items 4.5.1-4.5.7 and 4.4.1.

4.5.1 Determine if the reported structure can be located. If the structure cannnot be found, label the reported structure NL (place the label next to the sketch or in a column specifically designated for verified analyses).

4.5.2 If the reported structure is found, determine if a judgement can be made as to its countability. If the structure cannot be judged as to its countability due to beam damage, contamination or other factors, label the reported structure AMB.

4.5.3 If a judgement can be made as to the countability of the reported structure, determine if the structure is countable. If the reported structure is not countable, label it FP(number). A unique number is given to the FP label so that it can be specifically referred to in the report form. Optional: Check the other analyst's TEM analysis form. If the other analyst sketched the particle and correctly reported it as noncountable, label the particle TN(number). Note: The values for TN are not recorded on the report form.

4.5.4 If the reported structure is correctly identified as a structure, determine if it was reported as countable elsewhere on the same analyst's TEM analysis form (i.e., the analyst counted the structure twice). If it is a duplicate, label the reported structure FP(number).

4.5.5 If the reported structure is not a duplicate, label the structure TPU(number).

4.5.6 Determine if the other TEM operator recorded a sketch of the structure. If the other TEM operator did not report the structure on his/her TEM analysis form, place an FNB(number) on their TEM analysis form in the approximate location where the structure should have been found. The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.5.7 If the other TEM operator recorded a sketch of the structure, label the sketch with an FNA(number). The number should correspond to that given to the TPU on the first analyst's TEM analysis form.

4.6 Countable asbestos structures reported by neither TEM operator but found by the verifying analyst in the course of examining a grid square should be recorded on a separate TEM analysis form and labelled

TPV(number). The TEM operators should be assigned an FNA(number) or FNB(number) as described in items 4.5.6-4.5.7.

4.7 Complete the report form as described in items 4.7.1-4.7.10.

4.7.1 Complete the heading of the report form and fill in the initials or names of the two TEM operators on the first line of the report form table.

4.7.2 Count the number of asbestos structures obtained by each analyst and enter the value as SR (structures reported) on the report form.

4.7.3 Determine the number of true positives that are matched (TPM), the number of true positives that are unmatched (TPU) and the total number of true positives (TP) obtained for each TEM operator on the grid square and enter the values on the report form.

4.7.4 Determine and record on the report form the number of true positives found by the verifying analyst (TPV).

4.7.5 Determine and record on the report form the total number of structures (TNS) on the grid square.

4.7.6 Determine and record on the report form for each operator the following: 1) the number of false positives (FP), 2) the number of false negatives (FN), 3) the number of false negatives of type A and type B (FNA, FNB), 4) the number of structures that were not located (NL) and 5) the number of ambiguous structures (AMB).

4.7.7 Determine and record the values for TP/TNS, FP/TNS to two decimal places.

4.7.8 List on the report form the suspected reasons for the false positives obtained by each analyst. Some examples would be as follows: incorrect length measurement, structures counted twice, problem with interpretation of the counting rules, misidentification of a structure.

4.7.9 List on the report form the suspected reasons for false negatives (FNA and FNB). Some examples would be: incorrect length measurement, problem with interpretation of the counting rules, misidentification of material as asbestos, possible loss of sense of direction, and insufficient overlap of traverses.

4.7.10 Append any other relevant comments to the report form (quality of the preparation, etc.).

4.8 Check the numbers on the report form using the equations given in the calculation section.

5. Calculation

5.1 The values on the report form should be consistent with the following equations:

For both analyses:

$$TNS = TPM + TPU(\text{Operator 1}) + TPU(\text{Operator 2}) + TPV$$

For a given analysis:

$$SR = TP + FP + NL + AMB$$

$$TP = TPM + TPU$$

$$FN = FNA + FNB$$

$$TNS = TP + FN$$

$$1 = TP/TNS + FN/TNS$$

6. Precision and Bias

6.1 To determine the precision of the method, independent verified analyses were conducted by operators in two laboratories on a set of 21 grid squares. The mean value for TNS for the data set was 16.2 structures/grid square and the pooled standard deviation of the pairs of verified count determinations was 1.12 structures/grid square. The confidence at approximately the 95% level (2 standard deviations) of a reported verified count value in this data set is 2.24 structures/grid square or 13.9% of the mean value for TNS. We use 13.9% as an estimate of the imprecision of the method.

NOTE 6– The differences in the values obtained for the independent verified analyses described in item 6.1 are, for the most part, due to differences in interpretation of the counting rules. The structures analyzed in the study were complex and therefore the imprecision estimate discussed above likely represents an upper bound to the imprecision for the method.

6.2 The bias in the method will vary depending upon interpretation of the counting rules used in the analysis by the TEM operators and verifying analyst.

7. Keywords

7.1 asbestos; quality assurance; transmission electron microscopy; verified analysis

APPENDIXES

(Nonmandatory Information)

X1. TEST REPORT FORM

Fig. X1.1 The following format is suggested for use by the verifying analyst to report the comparison of the TEM operators' TEM analysis forms.

Grid box: _____ Date: _____

Grid slot: _____ Verifying Analyst: _____

Grid square: _____

	Analysis 1	Analysis 2
TEM Operator		
Structures Reported (SR)		
True Positives (TP)		
*TPM		
TPU		
*TPV		
*Total # Structures (TNS)		
False Positives (FP)		
False Negatives (FN)		
FNA		
FNB		
Not Located (NL)		
Ambiguous (AMB)		
TP/TNS		
FP/TNS		

*The values for these items will be the same for both analyses.

Test Report Form (continued)

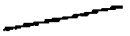
1) List details of suspected reasons for false positives. For each analyst describe reasons for FP1, FP2, FP3, etc. Note - it may not be possible to determine the reason for false positives for some structures.

2) List details of suspected reasons for false negatives (type A and type B). For each analyst describe reasons for FNA1, FNA2, etc.; FNB1, FNB2, etc. Note - it may not be possible to determine the reasons for false negatives for some structures.

X2. EXAMPLES OF COMPARISONS OF TEM ANALYSIS FORMS

[Note: The TEM analysis forms shown in the examples are abbreviated and do not contain analysis information. The AHERA counting rules (1987) were used for all analyses.]

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr

Analyst 2

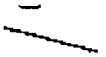
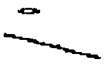
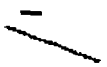
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.1 Example of matching structures on two TEM analysis forms (refer to item 4.3 of the procedure). Three structures on a grid square were found by both analysts. The relative order of the last two structures is different on the two TEM analysis forms; this may be due to the nature of the traverses by the analysts. Matching structures are indicated by TPM(number).

Analyst 1

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.7	0.1		TPM2	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		FP1	1	Chr

Analyst 2

Length (μm)	Width (μm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM3	1	Chr
0.7	0.1		TPM2	1	Chr

Fig. X2.2 Example of determining the status of an unmatched structure from TEM analysis forms (refer to item 4.4 of the procedure). Three of the structures match in the two analyses. The last structure of analyst 1 is unmatched but can be seen from the TEM analysis form to be a duplicate of the second structure obtained by the same analyst (the two structures have the same identification, dimensions, orientation and a similar nearby particle). The duplicate structure is therefore assigned an FP1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FNA1	0	Chr

Fig. X2.3 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the structure. However, analyst 2 has reported that the particle is not a structure (the cause of this oversight is not known). Analyst 1 is assigned a TPU1 and analyst 2 an FNA1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FP1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Fig. X2.4 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4 of the procedure). Both analysts have found the same particle as indicated by the dimensions, identification and orientation of the particle on both TEM analysis forms. However, analyst 1 has reported that the particle is a structure (the cause of this oversight is not known). Analyst 1 is assigned an FP1 and analyst 2 a TN1.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
1	0.6		TPM1 FNA1	1	Chr

Analyst 2

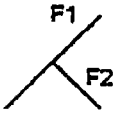
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
1	0.1	F1	TPM1	1	Chr
0.6	0.1	F2	TPU1	1	Chr

Fig. X2.5 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one countable structure and analyst 2 has reported two countable structures. Under the AHERA counting rules, analyst 2 is correct. The structure reported by analyst 1 is assigned both a TPM1 and an FNA1. The two structures reported by analyst 2 are assigned a TPM1 and a TPU1, respectively.

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

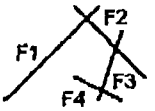
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

Fig. X2.6 Example of determining the status of unmatched structures from TEM analysis forms (refer to item 4.4.1 of the procedure). Both analysts have found the same asbestos-containing particle as indicated by the dimensions, identification, and orientation of the particle. However, analyst 1 has reported one structure and analyst 2 has reported four structures. Under the AHERA counting rules, analyst 1 is correct. The structure reported by analyst 1 is assigned a TPM1. The first structure reported by analyst 2 is labelled TPM1 and the remaining three reported structures are labelled FP1-FP3.

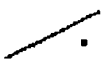
Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1			0	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1			1	Chr

a

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		FNA1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		TPU1	1	Chr

b

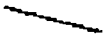
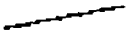
Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.4	0.1		TN1	0	Chr

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
0.6	0.1		FP1	1	Chr

c

Fig. X2.7 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same asbestos-containing particle as indicated by the identification and orientation of the fiber and by the presence of a similar particle nearby. However, the dimensions reported by the analysts differ and analyst 1 has reported zero structures and analyst 2 has reported one structure. The verifying analyst should determine the correct length of the fiber and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 2 is assigned a TPU1 and analyst 1 an FNA1. c) A second possible outcome is that the verifying analyst finds that analyst 2 is correct. Analyst 1 is assigned a TN1 and analyst 2 an FP1.

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1			1	Chr
1.0	0.1		TPM2	1	Chr

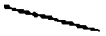
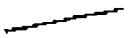
Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

a

Fig. X2.8 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Analyst 1 has reported one structure that analyst 2 has not reported. The verifying analyst should attempt to find the particle and determine if it qualifies as a structure. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is assigned a TPU1 and analyst 2 is assigned an FNB1. c) Another possible outcome is that the reported structure is not located. Analyst 1 is assigned an NL. Other possibilities (not illustrated) are that analyst 1 is incorrect (the particle is then labelled FP) or that the structure is too contaminated for characterization (the particle is then labelled AMB).

Analyst 1

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		TPU1	1	Chr
1.0	0.1		TPM2	1	Chr

Analyst 2

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		FNB1 TPM2	1	Chr

b

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
0.6	0.1		NL1	1	Chr
1.0	0.1		TPM2	1	Chr

Length (um)	Width (µm)	Sketch	Verification	# Structures	ID
1.3	0.1		TPM1	1	Chr
1.0	0.1		TPM2	1	Chr

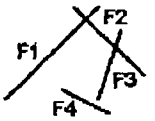
c

Fig. X2.8 (caption on previous page).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3			1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1		1	Chr
3	0.1	F2		1	Chr
2	0.1	F3		1	Chr
1	0.1	F4		1	Chr

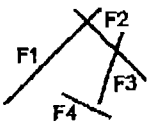
a

Fig. X2.9 Example of unmatched structures that must be examined by TEM (refer to item 4.5 of the procedure). a) Both analysts have likely found the same particle as indicated by the identification and orientation of the fibers. However, analyst 1 has recorded all fibers as touching (or intersecting) and has therefore counted the fiber arrangement as one structure under the AHERA method. Analyst 2 has reported four structures. The verifying analyst should find and examine the arrangement in the TEM to determine if the fiber labelled as F4 by analyst 2 is touching or intersecting the fiber labelled as F3. b) One possible outcome is that the verifying analyst finds that analyst 1 is correct. Analyst 1 is then assigned a TPM1 and analyst 2 is assigned a TPM1 and three FPs. Other possibilities (not illustrated) are that analyst 2 is correct (the structures reported by analyst 2 are then assigned a TPM and 3 TPUs and the structure reported by analyst 1 is assigned a TPM) or that the particle is too contaminated for identification (the structure reported by analyst 1 is then assigned a TPM and those reported by analyst 2 are assigned a TPM and three AMBs).

Analyst 1

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
5	3		TPM1	1	Chr

Analyst 2

Length (µm)	Width (µm)	Sketch	Verification	# Structures	ID
					
5	0.1	F1	TPM1	1	Chr
3	0.1	F2	FP1	1	Chr
2	0.1	F3	FP2	1	Chr
1	0.1	F4	FP3	1	Chr

b

Fig. X2.9 (caption on previous page)

X3. SUMMARY OF THE PROCEDURE FOR COMPARISON OF TWO TEM ANALYSIS FORMS

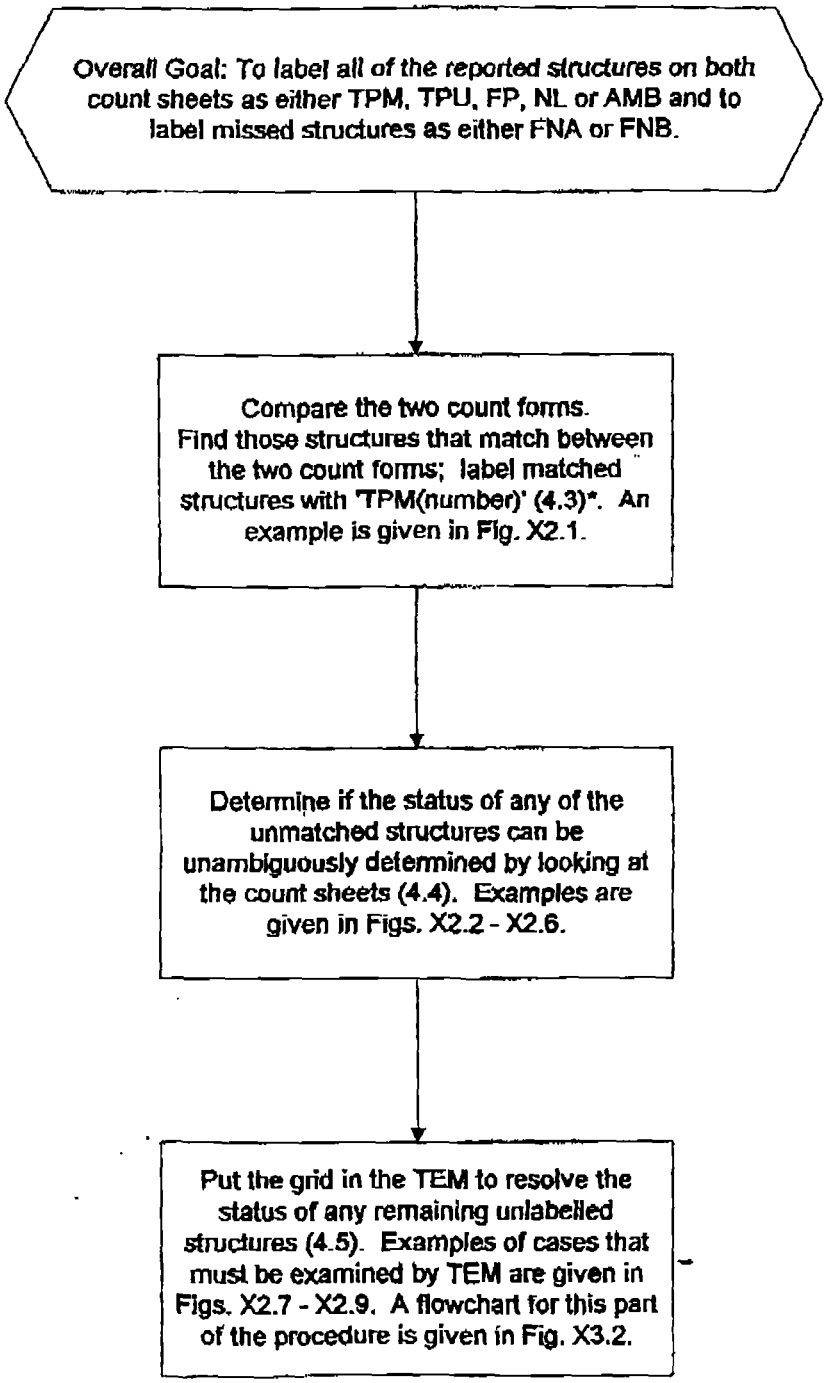


Fig. X3.1 Summary of the overall procedure for comparison of TEM analysis forms by the verifying analyst. *Numbers in parentheses in each block refer to the item number in the procedure.

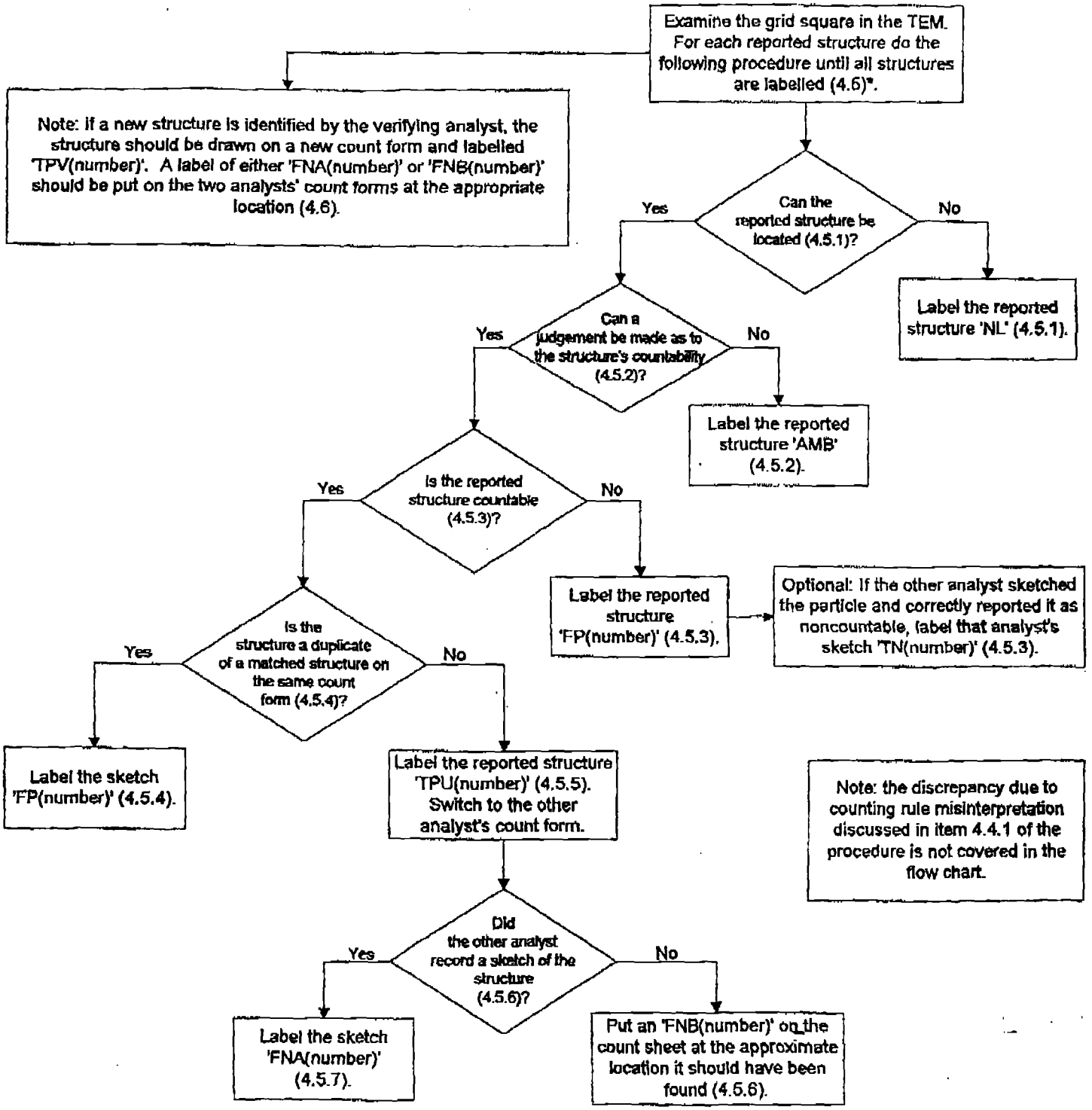


Fig. X3.2 Flowchart for examination of a structure in the TEM. The flowchart is an expansion of the last block in Fig. X3.1. *Numbers in parentheses in each block refer to the item number in the procedure.

ATTACHMENT 4

Statistical Comparison of Two Poisson Rates

1.0 INTRODUCTION

An important part of the Quality Control plan for this project is the repreparation and reanalysis of a number of TEM grids for quantification of asbestos fiber concentrations in air and dust. Because of random variation, it is not expected that results from repreparations samples should be identical. This attachment presents the statistical method for comparing two measurements and determining whether they are statistically different or not.

2.0 STATISTICAL METHOD

This method is taken from "Applied Life Data Analysis" (Nelson 1982). Input values required for the test are as follows:

N1 = Fiber count in first evaluation
S1 = Sensitivity of first evaluation
N2 = Fiber count in second evaluation
S2 = Sensitivity of second evaluation

The test is based on the confidence interval around the ratio of the two observed Poisson rates:

Rate 1 = N1 · S1
Rate 2 = N2 · S2
Ratio = Rate 1 / Rate 2

$$Lower\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1}{N2+1}\right) / F\left[\frac{1+\gamma}{2}; 2\cdot N2+2, 2\cdot N1\right]$$

$$Upper\ Bound = \left(\frac{S1}{S2}\right)\left(\frac{N1+1}{N2}\right)\cdot F\left[\frac{1+\gamma}{2}; 2\cdot N1+2, 2\cdot N2\right]$$

where γ is the confidence interval (e.g., 0.95) and F[δ; df1, df2] is the 100δth percentile of the F distribution with df1 degrees of freedom in the numerator and df2 degrees of freedom in the denominator.

If the lower bound of the ratio is > 1, then it concluded that rate 1 is greater than rate 2 at the 100(1-γ)% significance level. If the upper bound of the ratio is < 1, then it concluded that rate 1 is less than rate 2 at the 100(1-γ)% significance level. Otherwise, it is concluded that rate 1 and rate 2 are not different from each other at the 100(1-γ)% significance level.

Example:

N1 = 4 structures
S1 = 0.0001 $(cc)^{-1}$
Rate 1 = 4 · 0.0001 = 0.0004 s/cc

N2 = 6 structures
S2 = 0.001 $(cc)^{-1}$
Rate 2 = 6 · 0.001 = 0.006 s/cc

γ = 0.95

$$Lower\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4}{6+1}\right) / F\left[\frac{1+0.95}{2}; 2\cdot 6+2, 2\cdot 4\right] = 0.014$$

$$Upper\ Bound = \left(\frac{0.0001}{0.001}\right)\left(\frac{4+1}{6}\right) \cdot F\left[\frac{1+0.95}{2}; 2\cdot 4+2, 2\cdot 6\right] = 0.281$$

In this example, because the upper bound of the ratio is < 1, it is concluded that Rate 1 (0.0004 s/cc) is less than Rate 2 (0.006 s/cc) at the 95% significance level.

3.0 REFERENCES

Nelson W. 1982. Applied Life Data Analysis. John Wiley & Sons, New York. pp 438-446.

ATTACHMENT 5

NVLAP Airborne Asbestos Proficiency Test 98-2: Grid Orientation

Instructions for Form 1

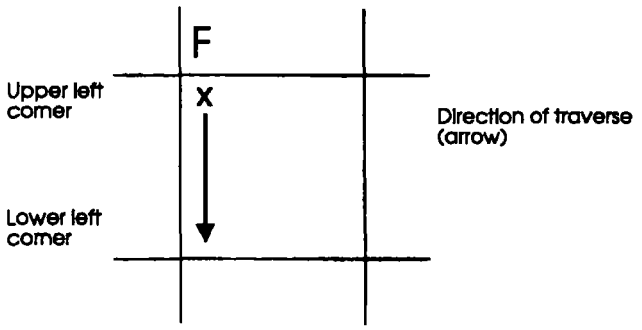
The following procedure is designed to ensure that all laboratories count the grid squares in the same orientation and scan direction to allow for verified analyses which will be performed in the next round of proficiency testing.

1. Put a grid into the TEM. Find a particle at the magnification typically used for asbestos analysis. Move the particle using one stage translation and record the direction of movement of the particle on *Form 1*. Move the particle using the other stage translation knob and record the direction of movement. Recording the two directions of movement should roughly form a cross. The cross represents the translation directions of your microscope at the magnification used for asbestos analysis. ***Draw the letter "F" onto the cross so the sides of the letter are parallel to the translation directions and the letter is upright and is not inverted.*** See the example on *Form 1*.

2. Decrease the magnification and locate the letter "F" on the finder grid. Increase the magnification of the TEM to that typically used for asbestos analysis by your lab, keeping the letter "F" in the field of view. Compare the orientation of the "F" to the cross drawn in step 1. If the letter "F" is not oriented as shown in your sketch, remove the specimen holder and rotate or invert the grid as necessary to correctly align the grid. This may require several iterations.

3. When the correct orientation is found, record the grid's position in the specimen holder as shown in the example of the second part of *Form 1*. Indicate in your drawing where the straight side and the notched portion of the grid are located. All grids analyzed in this proficiency test should be oriented in the same manner (always check that the letter "F" is in the correct orientation and that the X-Y translation directions allow translation roughly parallel to the grid bars).

4. The starting point of the traverse for structure counting must correspond to the upper left corner on the grid square. The "X" marks the starting corner of the traverse (your grid square may be at an angle to that shown in the example):



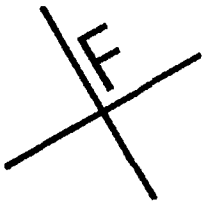
The initial direction of traverse must be from the upper left corner to the lower left corner of the grid square. If correctly oriented, the edge of the grid bar will remain in the field of view during the entire initial traverse (some allowance must be made for curvature or irregularly shaped grid bars.) If the grid is not oriented properly, go back to step 2.

NVLAP Lab Code: _____

Form 1. ***Grid Orientation***

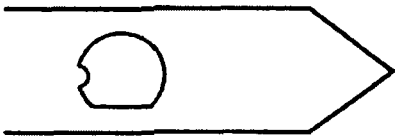
1. Sketch the orientation of the X-Y translation directions of the electron microscope as projected onto the electron microscope stage. Record the letter "F" as shown in the example below:

EXAMPLE:



2. Sketch below the orientation of the grid relative to the sample holder as shown in the example below:

EXAMPLE:



ATTACHMENT 6

Grid Opening Template for Sketching the Relative Position of Observed Structures

STRUCTURE LOCATIONS WITHIN GRID OPENING

****NOTE: Sketches only need to be completed for interlab analyses and repreps associated with interlabs*

Lab Name: _____ Lab Job Number: _____

Index ID: _____ Lab Sample ID: _____

Lab QC Type (circle one): Reprep for interlab Interlab

Grid: _____ Grid Opening: _____

upper left corner

traverse direction

Comments: